

# The Journal *of the* Society of Dyers and Colourists

Volume 77



Number 1

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The methods of fastness testing sponsored by the Society of Dyers and Colourists are published as a separate handbook under the title "Standard Methods for the Determination of the Colour Fastness of Textiles", the first edition appearing in 1955.

Developments in fastness testing, particularly internationally, necessitated the publication in 1958 of a supplement to the handbook and since then further developments have occurred. Instead of issuing a second supplement, however, the Society's Fastness Tests Co-ordinating Committee decided that it would be more convenient for the Society to issue a Second Edition of Standard Methods for the Determination of the Colour Fastness of Textiles which is now available from the offices of the Society.

The Second Edition contains the tests, etc., which appeared in the *Journal* as Sponsored Publications since the supplement was published—

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To facilitate ease of reference, the tests have now been arranged alphabetically and a standard lay-out has been adopted.

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(Abstracts section only—see page 2)

## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1-8 of the January 1961 and pages 389-396 of the July 1960 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURES

The Biological Chemistry of Cellulose

*J. A. Gascoigne*

New Observations on the Tippy Dyeing of Wool

*H. R. Hadfield and D. R. Lemin*

Dyeing and Printing Polyester and Polyester-Cellulosic Fabrics with Polyestren Dyes *H. Musshoff*

### COMMUNICATIONS

The Absorption of Saturated Azobenzene Vapour by Cellulose Acetate

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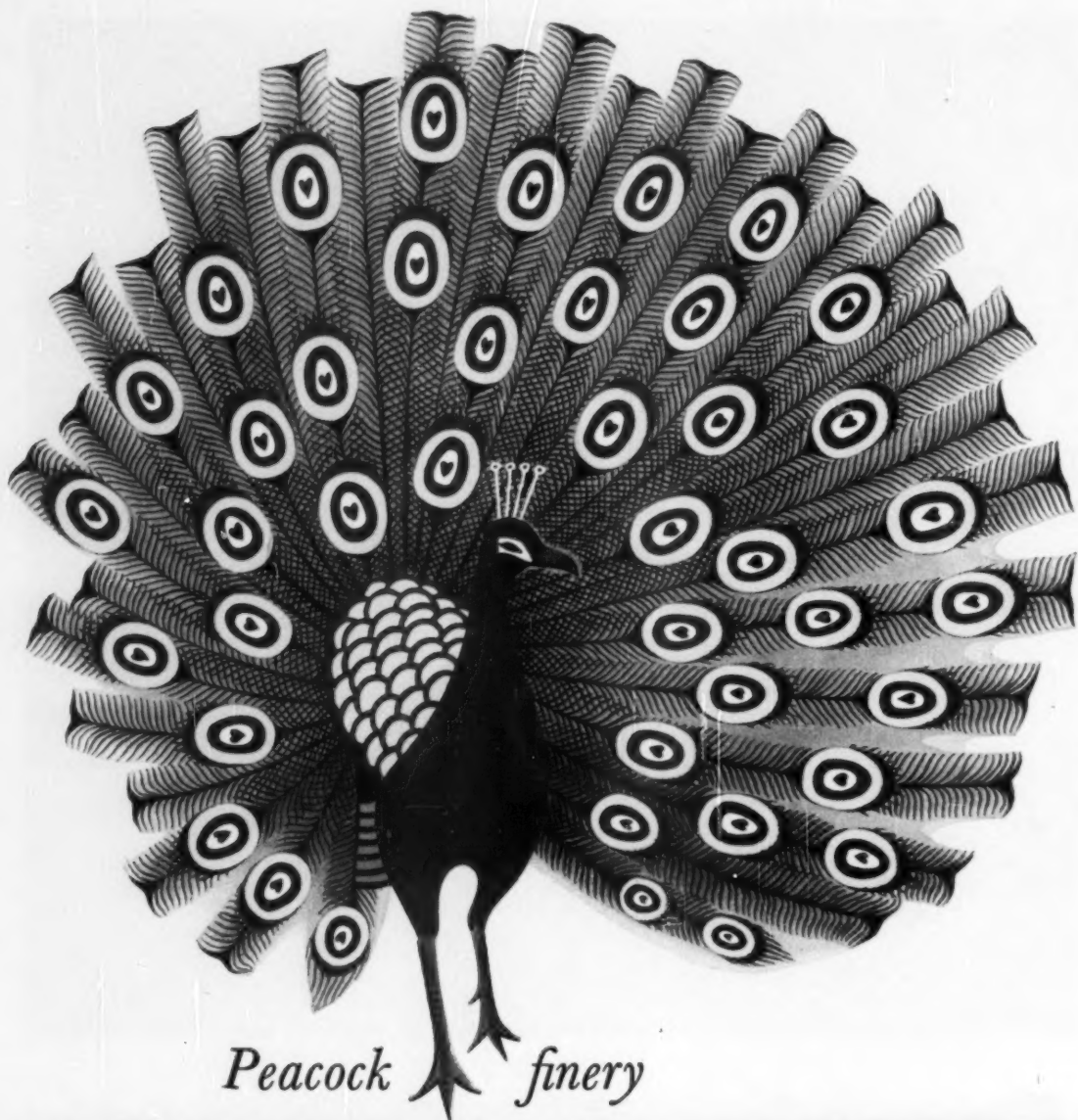
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# THE JOURNAL

OF THE

# Society of Dyers and Colourists

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Volume 77 Number 1

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Issued Monthly

## THE SOCIETY OF DYERS AND COLOURISTS

The Society of Dyers and Colourists was founded in Bradford in 1884, with the object of "promoting Scientific and Technical Knowledge among Textile Colourists, and the general advancement of the interests of the tinctorial and allied industries excluding all questions connected with wages and trade regulations."

The Society has eight Sections—Huddersfield, London, Manchester, Midlands, Northern Ireland, Scottish, West of England and South Wales, and West Riding—with four Junior Branches—Bradford, Leeds, Manchester, and Scottish. It has two affiliated societies—The Society of Dyers and Colourists of Australia and the Dyers and Colourists Association of South Africa.

### Objects

To promote the advancement of science and technology, especially in the theory and practice of the tinctorial arts.

To provide means for the wider dissemination and interchange of knowledge concerning the science and technology of colour and colouring matters, of their methods of application, and of the materials to which they may be applied.

To encourage education and research in any or all of the above subjects.

And for the furtherance of these objects—

To hold meetings for the reading of papers, for lectures, and for discussions.

To publish a *Journal*.

And to do all such other things as may be conducive to the attainment of these objects.

### Election of Officers and Members of Council

Officers and Members of Council—other than the President and President-elect, who are nominated by Council and elected at the Annual General Meeting—are elected by a Postal Ballot of the members.

The attention of members is drawn to Bye-laws No. 12–21 inclusive and 22–26 inclusive, which relate to Officers and Members of Council respectively.

Nominations must be received by the Honorary Secretary of the Society six weeks prior to the date of the Annual General Meeting.

### Members and Junior Members

Persons desirous of joining the Society as Ordinary Members or Junior Members may obtain Application Forms from the Society's offices or from any Section of the Society. Applications must be proposed and seconded by members to whom the applicants are known personally. The rates of annual subscription are as follows—

- (i) Ordinary Members—£4 4s. 0d.
- (ii) Junior Members (under 21 years of age)—£1 1s. 0d.
- (iii) Ordinary Members between the ages of 21 and 25 years who are students, i.e. who are pursuing an accepted course, full or part time, at a recognised technical college or university and vouched for by the head of their department or other responsible person—£1 1s. 0d.
- (iv) Other Ordinary Members between the ages of 21 and 25 years—£2 12s. 6d.
- (v) Members having had continuous membership of the Society for forty years to be given the option of paying half the current annual subscription for Ordinary Members.
- (vi) Members having had continuous membership of the Society for fifty years—the annual subscription to be waived entirely.
- (vii) During the period of national service the annual subscription may be waived. Such members will be given the opportunity of continuing to receive the *Journal* without charge.

### ***Diplomas in Tinctorial Technology***

The Society confers diplomas in tinctorial technology on suitably qualified members. These distinctions are in two grades—the Associateship (A.S.D.C.) and the Fellowship (F.S.D.C.). To obtain the Associateship candidates are required to pass the prescribed examinations and to furnish evidence of satisfactory general education and of training and experience in at least one branch of the manufacture or application of colouring matters. The Associateship has been accepted as a degree equivalent for the purposes of the Burnham Main and Technical Reports. The Fellowship is conferred on senior members who have attained high standing in the knowledge and practice of tinctorial technology. Students who intend to take the Society's examination are strongly recommended to become Registered Students, so that their studies and preparation may be directed effectively.

Copies of the regulations and syllabuses may be obtained on application to the offices of the Society.

### ***The Journal***

All members of the Society receive a copy of the *Journal* monthly.

Subscribers may receive the *Journal* for twelve months on payment of £7 0s. 0d.

Back numbers are supplied at 11s. 8d. per copy.

Communications on any subject related to the objects of the Society, especially such as are of an original character, are invited for consideration for publication in the *Journal*. Such Communications in the first instance should be addressed to the Editor at the offices of the Society.

General communications, including enquiries or orders for advertisements, should be addressed to the General Secretary.

### ***Reprints of Lectures and Communications***

Reprints of all lectures and communications are available if ordered immediately after publication. Further details are obtainable from the offices of the Society.

### ***Abstracts Section***

One-sided copies of the Abstracts, suitable for pasting on cards, are available on application to the offices of the Society.

### ***Free Advertisements***

For the convenience of members a limited number of advertisements relating to SITUATIONS WANTED may be inserted in the *Journal* gratis. Such advertisements must not exceed 24 words in length.

Replies may be addressed *Box—, Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.*

### ***Notice to Authors of Papers***

The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor at the offices of the Society and should arrive, if possible, before the monthly meeting of the Publications Committee, which is normally held on the third Tuesday in each month. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum if authors submit two copies of the typescript.

In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, and well-known experimental methods should be described only very briefly.



Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. Centre headings should be employed sparingly. Side headings should be indented and underlined, and run into the text to which they apply by means of a dash. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer. Acknowledgments should be kept brief.

The list of references should be given at the end of the manuscript, and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the Index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Annual Index should be used (*see also* note on page 23). Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and results should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper, tracing paper or cloth, or, preferably, Bristol board (but duplicate drawings for refereeing purposes need be only roughly prepared). Graphs should be enclosed in a complete frame; co-ordinate lines should be omitted, except for very short lengths along the axes. Experimental points should be indicated by symbols selected from the following fourteen, which are readily available to the printer—

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In a series of Figures containing graphs of similar type, consistent use of symbols will be a help to the reader. In preparing drawings the author must see that thickness of and separation between lines and size of experimental points are adequate for reduction of the drawings, usually to column width (about 2½ in.). Any numbering or lettering on the drawings should be indicated lightly in pencil, not drawn in ink.

Twenty-five free copies of a reprint are supplied to the author of an original paper published in the *Journal*, or fifty free copies when there are two or more authors, and a further number may be purchased from the Society.

### Copyright

Original articles, papers, and communications printed in this *Journal* are copyright. Subject to full acknowledgment being made, up to, but not more than, 800 words may be published elsewhere, but application for permission to reprint *in extenso* should be addressed to the Editor of the *Journal* at the offices of the Society.

The Society subscribes to the Royal Society Fair Copying Declaration (J.S.D.C., 66, 54 (Jan. 1950); 67, 236 (June 1951)).

### The Society's Library

Many of the books and periodicals reviewed or abstracted in the *Journal* since 1948 (and in some cases earlier) are retained by the Society, and may be borrowed by members. Enquiries and applications should be directed to the Editor at the offices of the Society.

### Historical Records

The Society is glad to receive gifts of old books, pattern cards, and other documents for inclusion if suitable in its collection of historical records related to the tinctorial arts.

### Library of the Chemical Society—Loan of Books

Members of the Society may borrow books from the very extensive collection of works on pure and applied chemistry in the Library of the Chemical Society, by applying direct to *The Librarian, The Chemical Society, Burlington House, Piccadilly, London W.1 (REGent 0675-6)*, and referring to their membership of the Society of Dyers and Colourists.

### Deposit of Sealed Communications

The Society is prepared to receive from members and others and to keep as deposits Sealed Communications dealing with any subject relating to the theory or practice of the dyeing, printing, and kindred industries.

Further particulars will be supplied on application.

## MEDALS AND AWARDS

### Honorary Members of the Society

1886	Sir H. E. ROSCOE, M.P.	1934	H. GRANDAGE
1902	Dr. F. H. BOWMAN	1934	CHRISTOPHER RAWSON
1908	Prof. C. GRAEBE	1941	Prof. E. C. C. Baly, C.B.E.
1908	Prof. C. LIEBERMANN	1944	Prof. F. M. ROWE
1911	Prof. ADOLF VON BAEYER	1946	JAMES S. RIDSDALE
1914	Count HILAIRE DE CHARDONNET	1947	Dr. C. J. T. CRONSHAW
1917	Prof. ARTHUR G. GREEN	1948	H. JENNISON, M.C.
1919	R. VIDAL	1950	GEORGE E. HOLDEN, C.B.E.
1920	The Prime Warden of the Worshipful Company of Dyers ( <i>ex officio</i> )	1953	JOHN BARRITT, O.B.E.
1921	HORACE A. LOWE	1954	FRED SCHOLEFIELD
1923	CHARLES F. CROSS	1954	Dr. C. M. WHITTAKER
1925	MAURICE PRUD'HOMME	1955	Sir ROBERT ROBINSON, O.M., Nobel Laureate
1927	ERNEST HICKSON	1956	FRED SMITH
1928	Dr. R. E. SCHMIDT	1959	H. H. BOWEN
1931	Dr. ALFRED RÉE	1959	W. H. CADY
1932	Prof. W. M. GARDNER	1959	CLIFFORD PAINE
1934	GEORGE DOUGLAS		

The following have been *ex officio* Honorary Members of the Society, the later dates indicating termination or change in the title of the office—

1886-1920	The Worshipful Master of the Dyers' Company	1900-1905	W. E. B. PRIESTLEY, Chairman of the Technical Instruction Committee of the Bradford City Council
1886-1900	The President, Bradford Tech- nical College		



### The Perkin Medal

The Perkin Medal is an excellent representation of the head of Sir William Perkin, the founder of the coal-tar colour industry, and President of the Society in 1907.

This medal is awarded for discoveries or work of outstanding importance in connection with the tinctorial arts.

### List of Recipients

1908	Professors GRAEBE and LIEBERMANN	Synthesis of Alizarin
1911	Prof. ADOLF VON BAEYER	Synthesis of Indigo
1914	Comte HILAIRE DE CHARDONNET	Artificial Silk
1917	Prof. A. G. GREEN	Primuline
1919	R. VIDAL	Sulphur Black
1921	HORACE LOWE	Permanent Lustre on Cotton
1923	C. F. CROSS	Discovery of Viscose
1925	M. PRUD'HOMME	Aniline Black and Alizarin Blue
1928	Dr. R. E. SCHMIDT	Anthraquinone Derivatives and Dyes
1938	Dr. H. DREYFUS	Development of Cellulose Acetate Industry
1938	J. BADDILEY	Services to British Dyestuffs Industry
1950	Prof. J. B. SPEAKMAN	Contributions to Science and Technology of Textiles
1954	Dr. A. ZITSCHER	Development of New Azoic Dyes
1956	Dr. W. H. CAROTHERS (posthumous award)	Discovery of Nylon
	JOHN R. WHINFIELD, C.B.E.	Discovery of Terylene
1959	Dr. C. J. T. CRONSHAW	As a Leader under whose Enthusiastic Guidance the Phthalocyanine Pigments and derived Textile Dyes were first made available and their Basic Constitution established
	J. T. MARSH	Outstanding Contributions to the Practice and the Literature of Textile Chemistry



### The Medal of the Society of Dyers and Colourists

This Medal was instituted by the Society in 1908. The design indicates that the work of the dyer (centre figure) is both a science (right background) and an art (left foreground).

1908-1927 The Medal was occasionally awarded as a recognition of work of exceptional merit carried out under the Society's Research Scheme.

From 1928 The Medal was awarded as a recognition of exceptional services (a) to the Society or (b) in the interests of the Tintorial and Allied Industries.

#### List of Recipients\*

##### Gold Medal

1928	ERNEST HICKSON	1948	G. G. HOPKINSON
1930	ARTHUR SILVERWOOD	1949	S. M. NEALE
1933	Prof. WALTER M. GARDNER	1950	P. W. CUNLIFFE
1934	Prof. F. M. ROWE		H. FOSTER
1940	F. L. GOODALL	1951	W. KILBY
1943	C. M. WHITTAKER	1953	H. A. TURNER
1946	H. H. BOWEN	1954	G. S. J. WHITE
	H. H. HODGSON	1955	P. W. CUNLIFFE (Bar)
1947	FRED SMITH	1956	C. O. CLARK
	F. SCHOLEFIELD		
1957	J. BARRITT, O.B.E.		

Exceptional Services to the Society as Chairman of the Colour Index Editorial Panel

1958	H. BLACKSHAW	}	Exceptional Services to the Society in Connection with the Colour Index
	H. H. BOWEN (Bar)		
	Prof. W. BRADLEY		
	C. O. CLARK (Bar)		
	J. G. GRUNDY		
	R. J. HANNAY		
	H. H. HODGSON (Bar)		
	M. LAPWORTH		
	A. THOMSON		
	T. VICKERSTAFF		

1959	I. D. RATTEE
	W. E. STEPHEN
	H. RIS

1960	W. A. EDWARDS
	W. PENN

Exceptional Services to the Society and for Research in the Tintorial Arts  
Invention and Development of the first Practical System of colouring Cellulosic Fibres with Reactive Dyes  
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Exceptional Services to the Society  
Exceptional Services to the Society and to the Tintorial and Allied Industries

##### Silver Medal

1912	J. H. GARNER	1947	C. SCHARDT
1934	L. A. LANTZ	1948	C. O. CLARK
	S. G. BARKER		D. B. F. McANDREW
	H. H. BOWEN	1950	L. A. LANTZ (Bar)
	P. W. CUNLIFFE	1953	A. W. CARPENTER
	R. S. HORSFALL	1954	C. C. WILCOCK
	Prof. B. A. McSWINEY	1955	H. W. ELLIS
	C. C. N. VASS		J. G. GRUNDY
	C. M. WHITTAKER		K. McLAREN
1936	W. A. EDWARDS	1955	J. PORTER
1937	R. RITCHIE		M. E. PROBERT
1946	Mrs. E. CUMMINGS (née Levin)		J. V. SUMMERSOILL
	E. RACE		E. WILSON
	H. TURNER	1957	F. ATACK
1958	A. W. CARPENTER (Bar)		
	R. L. ELLIOTT		
	H. TURNER (Bar)		
1959	G. G. BRADSHAW		
	H. F. A. BRASSARD		
	N. HAMER		
	J. W. REIDY		

Continued Valuable Services to the Society  
Valuable Services to the Society  
Continued Valuable Services to the Society  
Valuable Services to the Society and particularly as a Member of the Fastness Tests Co-ordinating Committee and the Publications Committee  
Valuable Services to the Society and for the Stimulation he has afforded the Society in many Aspects of its Work  
Valuable Contributions in Service to the Society  
Valuable Contributions in Service to the Society

\* Full citations of the earlier awards are given in J.S.D.C., 76, 5-7 (1960)

**Silver Medal—continued**

1960 R. GRICE  
E. I. NOBLE  
C. B. STEVENS

Valuable Services to the Society  
Valuable Services to the Society, especially in Australasia  
Valuable Services to the Society

**Bronze Medal**

1908 J. B. FOTHERGILL  
1958 Mrs. J. M. FIRTH  
K. MELDRUM  
J. K. SKELLY  
H. W. TAYLOR  
1959 W. R. LEIGH  
J. RAYMENT  
1960 F. HARRISON

1953 Miss M. FORBES H. R. HADFIELD  
Valuable Services to the Society  
Valuable Services to the Society  
Valuable Services to the Society  
Valuable Services to the Society  
Enthusiastic Services in the Interests of the Society  
Enthusiastic Services in the Interests of the Society  
Valuable Services to the Society since 1947 as Honorary Treasurer of the London Section



**The Worshipful Company of Dyers  
Research Medal**

The Medal represents the Arms of the Worshipful Company of Dyers of the City of London, which were granted in 1471. The following is a brief description—

*Arms*—Sable, a chevron engrailed argent, between three bags of madder of the last, corded or.

*Crest*—On a wreath three sprigs of the gaintree erect vert, fructed gules.

*Supporters*—Two leopards rampant gardant argent, spotted with various colours; fire issuing from their ears and mouth proper, both ducally crowned or.

*Motto*—Da Gloriam Deo.

1—The Dyers' Company offer annually a Gold Medal called "The Worshipful Company of Dyers Research Medal", the award of which is open to the authors of papers embodying the results of scientific research or technical investigation connected with the tinctorial arts submitted to the Society of Dyers and Colourists, and published in the *Journal* of such Society during the twelve months ending on the 30th June in the year for which the Medal is granted, and, in the special circumstance provided for by Rule 6, during the twelve months ending on the 30th June in the year previous to that for which the Medal is granted. If a paper shall be published in two or more parts, then for the purpose of the award of the Medal, all the parts together shall be treated as a paper published in the year in which the final part is published.

2—The Medal will not be awarded to the same person on more than one occasion.

3—The Society of Dyers and Colourists shall consider the papers available for the Medal and advise the Company as to the merits thereof, and if, in the judgment of the Society, none of the papers is of sufficient merit, the Society may recommend that the Medal be not awarded.

4—The Dyers' Company will award the Medal either to the author of the paper which, in all the circumstances, appears to the Company to show the greatest merit, or, in the event of such paper being the work of an author who has already been awarded the Medal, to the author of the paper next in order of merit who has not already been awarded the Medal, and may, if the Company so thinks fit, refrain from making any award.

5—In the event of a paper being the work of two or more persons, the author shall be taken to be that one of them whose work in the opinion of the Society of Dyers and Colourists has most substantially contributed to the merit of the scientific research or technical investigation embodied in such paper.

6—In the event of the author of a paper of sufficient merit published in the *Journal* of the Society of Dyers and Colourists during any twelve months for which the Medal is awarded being unsuccessful in obtaining the award of the Medal for that period, the Society of Dyers and Colourists may, in their discretion, consider such paper for adjudication with the papers available for the award of the Medal for the next succeeding twelve months.

**List of Recipients**

1908	Prof. E. KNECHT	Estimation of Degree of Mercerisation of Cotton
1909	Prof. A. G. GREEN	Chemical Technology of Aniline Black
1910-11	R. L. TAYLOR	Action of CO <sub>2</sub> and Air on Bleaching Powder
1911-12	W. HARRISON	Electrical Theory of Dyeing
1912-13	S. H. HIGGINS	Cotton Bleaching
1913-14	W. JOHNSON (in conjunction with Prof. A. G. Green)	Constitution of Aniline Blacks
1914-15	M. FORT	Mechanism of the Acid Dyebath
1915-16	J. R. HANNAY	Action of Copper on some Thiazine, Oxazine and Azine Dyes



**The Worshipful Company of Dyers Research Medal—continued**

1916-17	Prof. H. M. DAWSON	Acid Catalysis and Theory of Acids
1917-18	L. G. RADCLIFFE	Sulphonation of Fixed Oils
1918-19	C. F. CROSS (Diplomas to M. C. Lamb and C. V. Greenwood)	Colloidal Tannin Compounds
1919-20	A. E. EVEREST (Diploma to A. J. Hall)	Tinctorial Properties of Anthocyanins
1920-21	Prof. G. T. MORGAN	Co-ordination Theory of Valency
1921-22	S. JUDD LEWIS	Fluorescence of Cellulose and Derivatives
1922-23	Prof. ARTHUR G. GREEN (Bar) and K. H. SAUNDERS	The Ionamines
1923-24	S. JUDD LEWIS (Bar)	Determination of Fluorescence of Cellulose
1924-25	Prof. F. M. ROWE (Diploma to Miss E. Levin)	Identification of Azoic Dyes and Pigments
1925-26	H. H. HODGSON	Structure of Sulphide Dyes
1926-27	Prof. F. M. ROWE (Bar) (Diplomas to Miss E. Levin, A. C. Burns, J. S. H. Davis and W. Tepper)	Preparation of Phthalazine, Phthalazone and Phthalimide Derivatives
1928-29	F. SCHOLEFIELD (Diplomas to Miss E. Hibbert and C. K. Patel)	Action of Light on Vat-dyed Cotton
1929-30	H. H. HODGSON (Bar)	Colour and Constitution—Electronic Theory
1930-31	Prof. F. M. ROWE (Second Bar) (Diplomas to S. Ueno and F. H. Jowett)	Action of Boiling Caustic Soda on Azoic Dyes
1932-33	W. T. ASTBURY J. B. SPEAKMAN	X-Ray Interpretation of Fibre Structure Structure of Wool Fibre
1934-35	H. A. TURNER (Diplomas to G. M. Nabar and F. Scholefield)	Effect of Reduced Vat Dyes on Cellulose Oxidation
1935-36	Prof. F. M. ROWE (Third Bar) (Diplomas to C. H. Giles, R. L. M. Allen, W. G. Dangerfield, and Glyn Owen)	Decomposition of Azoic Dyes
1936-37	J. B. SPEAKMAN (Bar) (Diplomas to C. S. Whewell and J. L. Stoves)	Reactivity of Sulphur Linkage in Animal Fibres
1937-38	Prof. F. M. ROWE (Fourth Bar) J. B. SPEAKMAN (Second Bar) (Diplomas to E. Race and T. Vickerstaff)	Unlevel Dyeing of Wool with Acid and Chrome Dyes
1939-40	T. H. MORTON	Application of Vat Dyes to Viscose Rayon
1941-42	T. VICKERSTAFF	Dyeing Cellulose Acetate with Disperse Dyes
1943-44	J. BOULTON	Importance of Dyeing Rate
1946-47	H. PHILLIPS	Constitution and Properties of Bisulphited Wool
1947-48	J. M. PRESTON	Dyeing of Viscose Rayon; Heating and Drying of Textiles
1948-49	H. LINDLEY	Set and Supercontraction in Wool
1950-51	J. CRANK	Diffusion of Direct Dyes into Cellulose
1951-52	H. HAMPSON	Application of Vat Dyes to Viscose Rayon Cakes
1952-53	R. H. PETERS	Reduction Properties of Vat Dyes
1953-54	R. J. HANNAY	pH Control in Metachrome Dyeing
1954-55	C. H. GILES	Brazilwood and Logwood—Fibre Adsorption and Lakes
1955-56	C. L. BIRD	Dyeing Cellulose Acetate with Disperse Dyes
1956-57	K. McLAREN	Light-fastness Testing and Fading
1957-58	A. JOHNSON as senior author	Series of papers on The Chemistry of Esters of Leuco Vat Dyes
1959-60	J. WEGMANN	Author of paper on Effect of Structure on the Change in Colour of Vat Dyes on Soaping

### ***The Worshipful Company of Feltmakers Research Medal***

The Worshipful Company of the Art or Mystery of Feltmakers of London offer annually a medal to the author of a paper dealing with the properties of wool, fur or other felting fibres which contributes to the greater understanding of the process of felting, of feltmaking, or the coloration of felts, and which is published in the *Journal of the Society of Dyers and Colourists*.

### ***Knecht Memorial Fund***

A Fund of £100 was subscribed as a Memorial to the late Professor Edmund Knecht. From the interest on this sum two prizes, in the form of books, are awarded annually to selected students of the Manchester College of Science and Technology and the Royal Technical College, Salford.

### ***Mercer Lecture***

This lecture was instituted by an anonymous donor in commemoration of the centenary of the discovery of mercerisation.

1944	N. G. McCULLOCH and G. S. HIBBERT	Science in an Old Industry
1945	G. S. J. WHITE and T. VICKERSTAFF	Colour
1946	D. ENTWISTLE	Regenerated Fibres from Natural Polymers
1947	E. J. BOWEN	Colour and Constitution—The Absorption of Light by Chemical Compounds
1948	F. FARRINGTON	Textile Printing
1949	E. WILSON	Some Applications of Chemistry to Textile Finishing
1950	A. B. MEGGY	Some Recent Developments in the Theory of Dyeing
1951	D. TRAILL	Some Trials by Ingenious Inquisitive Persons—Regenerated Protein Fibres
1952	F. C. WOOD	Non-felting Wool and Wool Mixtures
1953	J. R. BLOUKEY and D. H. TUCK	The Coloration of Leather
1954	N. W. YELLAND	Physics in the Dyeing, Printing, and Finishing Industries
1955	G. LANDELLS	Modern Resin Finishing
1956	R. K. FOURNESS	Disperse Dyes—Their Development and Application
1957	J. S. WARD	The Influence of Fibre Types on Dyeing Methods
1959	I. S. MOLL	Aspects of Pigment Dispersion related to Usage

### ***George Douglas Lecture***

A sum of £2000 was given to the Society by the Bradford Dyers' Association Ltd., the income from the investment of which is employed in providing a biennial lecture on some subject connected with the colouring or finishing of textiles.

1949	H. LEVINSTEIN	George Douglas, His Times, and Some Thoughts on the Future
1952	R. HILL	Synthetic Fibres in Prospect and Retrospect
1953	T. HOLBROOKE	The Search for New Dyes in relation to Modern Developments in the Textile Field
1957	G. S. J. WHITE	Dyeing—The Apotheosis of Synthesis or a Team playing for its Colours against White
1958	W. GUTMANN	Textile Finishing—Problems of Today and Tomorrow
1960	A. B. D. CASSIE	Wool as a Fibre in Dyeing and Finishing

## COMMUNICATIONS

## The Adsorption of Non-ionic Dyes by Cellulose

J. R. ASPLAND and C. L. BIRD

Linear isotherms have been obtained on viscose rayon, in the range 20–90°C, with benzidine, *o*- and *m*-tolidine, and derivatives of aminoazobenzene. *o*-Tolidine has much higher affinity for cellulose than the non-coplanar *m*-tolidine. Heats of dyeing were found to be constant between 40 and 80°C. Non-ionic dyes do not appear to form aggregates in aqueous solution.

## INTRODUCTION

The aim of this work was to complete a study of the behaviour of non-ionic dyes on various substrates. The characteristic linear adsorption isotherm had been found on wool<sup>1</sup>, and it appeared probable that similar results would be obtained with cellulose. It was hoped that suitable non-ionic dyes resembling direct dyes could be obtained by tetrazotising benzidine and coupling with 2 mol. of (a)  $\alpha$ -naphthylamine and (b) *NN*-di( $\beta$ -hydroxyethyl)aniline. These dyes were prepared, but after purification (m.p. 249 and 278°C, respectively) they were found to be completely insoluble in hot water and could not be used in dyeing experiments, even in presence of dispersing agent. It was therefore necessary to use disperse dyes, some of which stain cellulose, although much less heavily than wool. It was also decided to include benzidine, and *o*- and *m*-tolidine, in order to study the effect of coplanarity.

Curcumin (C.I. 75300) was examined, but was found to be unstable. However, one non-ionic compound with some affinity for cellulose and slight aqueous solubility was found in the form of a fluorescent brightening agent.

No previous work on the adsorption of non-ionic dyes by cellulose from water appears to have been carried out, but Lamparsky and Rack<sup>2</sup> have examined the partition between cotton and water, at 55°C and pH 5.5, of amino- and hydroxy-compounds, e.g. 4,4'-diamino- and 4,4'-dihydroxy-diphenylmethane. These workers obtained approximately linear isotherms in eight cases and non-linear isotherms in four cases; partition coefficients were approx. 2–5.

## EXPERIMENTAL

The cellulosic fibre used was Courtaulds' 150/27 continuous-filament viscose rayon. It was Soxhlet-extracted with 40–60°C ligroin, dried, washed thoroughly with distilled water at 60°C, dried, and conditioned at 65% r.h. and 22°C. A few experiments were carried out, after conditioning, with purified Egyptian Giza 12 cotton yarn, kindly supplied by the Shirley Institute.

Most of the dyes were available from previous work. The fluorescent brightening agent (X) was obtained from the commercial product by recrystallising three times from 64 O.P. ethyl alcohol. Benzidine of analytical reagent grade was used. *o*-Tolidine was obtained from the commercial product by rapid distillation *in vacuo*, followed by rapid recrystallisation from 64 O.P. ethyl alcohol containing a little activated charcoal. *m*-Tolidine was obtained from a recrystallised sample of the hydrochloride. This was dissolved

in water, ammonia gas was bubbled through the solution, and a white tar, which solidified on standing, was obtained. It was recrystallised from water, care being taken to prevent formation of a colourless oil on heating. The product was slightly hygroscopic and was stored in a vacuum desiccator.

Dyeings were carried out in a Marney machine fitted with a propeller stirrer in the water bath. The temperature in the dye vessels was thermostatically controlled to within  $\pm 0.25$  deg.C. Viscose rayon hanks were attached to formers constructed from stainless steel wire and fastened to the reciprocating arms of the machine. The dyeing vessels were 110-ml Pyrex tubes (8 in.  $\times$  1 in.) supported by corks in the top plate of the machine. The formers passed through small water-condensers (2-in. high), which fitted into rubber bungs at the top of the dyeing vessels.

Initially, approximately saturated solutions were made up at the dyeing temperatures, and differing amounts of these solutions were placed in the dyeing tubes, with sufficient distilled water to bring the total volume to 80 ml. After allowing 15 min for the dyebath to reach the temperature of the thermostat, approx. 1 g of conditioned viscose rayon yarn was introduced, giving a liquor:yarn ratio of 80:1. Dyeing was then continued for a predetermined time.

A slightly different method was used for obtaining dyeings up to saturation of the aqueous phase. When the partition coefficient was known, it was possible to calculate the amount of dye required to give a saturated solution after dyeing to equilibrium. A solution containing this amount of dye was prepared at a temperature above the dyeing temperature and added to the dyebath. A viscose rayon hank was then added and dyeing took place, the temperature gradually falling to that of the thermostat.

Preliminary experiments with several of the dyes showed that equilibrium was reached rapidly, 4 h being sufficient at 40°C and 2 h at 60°C. Where equilibrium times were not determined, 8 h at 40°C and 4 h at 60°C were allowed.

At the end of the dyeing, each hank was removed, squeezed, transferred to a stoppered bottle, and weighed. Dye was removed from the fibre by Soxhlet-extraction with 50% aqueous 64 O.P. alcohol. The cooled extract was made up to a known volume and the dye present was estimated spectrophotometrically in the usual way, allowance being made for entrained dye liquor. Dye in the residual dyebath was also estimated spectrophotometrically.

TABLE I

Dye No.	Chemical Formula	M.p. (°C)	-Δμ° (kcal/mole)			-ΔH° <sub>dye</sub> (kcal/mole)	ΔH° <sub>H<sub>2</sub>O</sub> (kcal/mole)	ΔH° <sub>fibre</sub> (kcal/mole)	-ΔS° (cal/mole per deg. C)
			40°C	60°C	80°C				
I		126	2.12	1.91	1.80	4.7	10.3 <sup>1</sup>	5.6	8.4
II		136	1.84	1.71	1.57	3.9	8.9 <sup>1</sup>	5.0	6.7
III		249	2.31	2.08	1.81	6.2	10.1*	3.9	12.3
IV		157	1.95	1.82	—	5.0	12.5	7.5	9.4
V		215	—	(2.85)	(2.49)	7.3	15.4 <sup>5</sup>	8.1	13.4
VI		206	—	2.48	2.28	5.9	16.9 <sup>6</sup>	11.0	10.2
VII		162	3.06 On cotton (3.14)	2.79	2.56	7.0	12.4*	5.4	12.7
VIII		171-172	—	3.23	2.87	9.3	15.9	6.6	18.3
IX		142	—	(3.16)	2.79	8.7	14.2 <sup>1</sup>	5.5	16.7
X		250-251	3.69	3.35	—	9.2	11.2	2.0	17.6
XI		127-128	—	1.55	(1.28)	6.3	11.2	4.9	14.3
XII		120	2.10	1.92	—	4.5	8.8	4.3	7.3
XIII		92	1.01	0.88	—	2.6	7.4	4.8	5.1

Additional values for -Δμ°: IV, 2.18 at 20.8°; V, 2.65 at 70° and 2.26 at 90°; VII (on cotton), 3.11 at 42°; IX, 3.00 at 70° and 2.66 at 90°; XI, 2.11 at 21.8° and 1.22 at 85°C.

Values in parentheses were obtained by extrapolation.

Bird and Harris<sup>5</sup> give 14.0 for III and 15.1 for VII.

Desorption experiments with VIII at 80°C and X at 60°C showed that the equilibrium was truly reversible.

When not already available, aqueous solubilities were determined in the usual way, over a range of temperatures between 20° and 90°C. From the linear plots of log (solubility) against 1/T, where T is the absolute temperature, heats of aqueous solution (ΔH°<sub>H<sub>2</sub>O</sub>) were obtained.

#### INTERPRETATION OF RESULTS

The adsorption isotherms showed a linear relation at each temperature up to saturation of the aqueous phase (as illustrated in Fig. 1), with all the compounds examined<sup>3</sup>. The straight lines (obtained by the method of least mean squares) do

not pass through the origin. This is due to the presence in the dye extracts of particles of viscose rayon which could not be removed by filtration or centrifuging and which absorbed visible and ultraviolet radiation. This absorption was found to vary with wavelength and with the time of extraction, but was reasonably constant for a particular series of dyeings extracted for the same length of time.

The partition coefficient, K, is given by the slope of the straight line—

$$K = ([D]_f - [D]_{f0})/[D]_s = [D]_f/[D]_s \dots (1)$$

where [D]<sub>f</sub> is the apparent concentration of dye on the fibre and [D]<sub>f0</sub> is the magnitude of the intercept on the vertical axis. [D]<sub>f</sub> is calculated



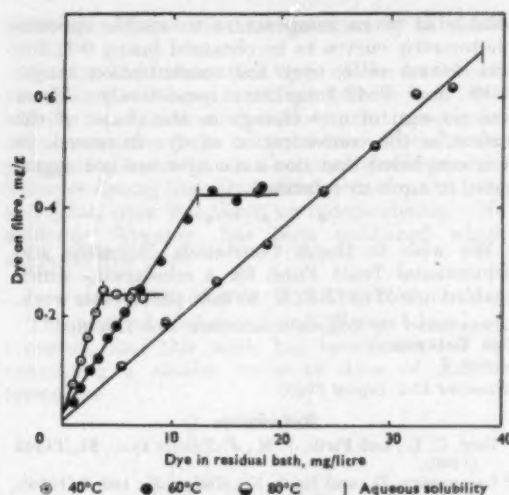


FIG. 1—Adsorption isotherms for dye VII

in terms of g dye per kg of oven-dry fibre and  $[D]$ , in terms of g dye per litre.

The standard affinity,  $\Delta\mu^\circ$ , is obtained from the equation—

$$-\Delta\mu^\circ = RT \ln K - RT \ln V \quad \dots (2)$$

where  $R$  is the gas constant,  $T$  is the absolute temperature, and  $V$  is the volume term (in litres per kg of dry fibre). The values taken for  $V$  are those used by Marshall and Peters<sup>4</sup>, namely, 0.30 and 0.45 litres/kg for cotton and viscose rayon, respectively.

The heat of dyeing,  $\Delta H^\circ_{\text{dye}}$ , is obtained from the slope of the linear plot of  $-\Delta\mu^\circ/T$  against  $1/T$ , and the entropy change,  $\Delta S^\circ$ , from the equation—

$$\Delta\mu^\circ = \Delta H^\circ_{\text{dye}} - T\Delta S^\circ \quad \dots (3)$$

Heats of solution in the fibre are obtained from the equation—

$$\Delta H^\circ_{\text{fibre}} = \Delta H^\circ_{\text{H}_2\text{O}} + \Delta H^\circ_{\text{dye}} \quad \dots (4)$$

The results obtained are given in Table I.

#### DISCUSSION

Affinities are seen to decrease in the usual way with increase in temperature. The values are low, owing to the small size of the molecules compared with those of direct dyes. The highest values are given by the largest molecules (VIII, IX, and X).

On comparing III with VIII, and II with IX, it is seen that at 60°C the increases in affinity resulting from the additional benzene ring are 1.15 and 1.45 kcal/mole, respectively. With II and IX on wool<sup>1</sup> the increase is 1.27 kcal/mole. One methyl substituent in a benzene ring (cf. VI and VII) increases the affinity by 0.31 kcal/mole, and two methyl substituents (cf. XI and XII) increase it by 0.37 kcal/mole. On the other hand, replacement of  $-\text{NH}_2$  by the larger but more hydrophilic  $-\text{N}(\text{C}_2\text{H}_5\text{OH})_2$  group decreases the affinity by 0.20 (I and II), 0.26 (III and IV), and 0.37 (V and VI) kcal/mole, respectively; on wool<sup>1</sup> the decrease with I and II is 0.26 kcal/mole.

Dyeing in presence of 0.05-N sodium chloride increased the affinity of *p*-aminoazobenzene (I) slightly (from 1.91 to 1.97 kcal/mole), and there

was a small decrease in aqueous solubility and fibre saturation value.

The values for VII on viscose rayon and cotton are sufficiently close to suggest that the affinity of non-ionic dyes is not affected by the type of cellulose (as found by Marshall and Peters<sup>4</sup> for direct dyes), or by the presence of carboxyl groups in viscose rayon.

The low affinity of *m*-tolidine (compared to that of *o*-tolidine) is noteworthy, and supports the accepted view that coplanarity of the dye molecule is essential for imparting affinity.

The very low affinity of the dyes is reflected in the very low fibre saturation values shown in Table II. The saturation values for VII and VIII, namely, <0.05%, indicate the maximum amount of staining of cellulosic fibres by disperse dyes likely to be experienced in practice. The saturation values of non-ionic dyes on cellulose acetate at 60°C appear to be roughly 20 times those on wool, and 50–100 times those on cellulose. On all fibres, saturation values increase with increasing temperature, and the highest values (as well as the lowest partition coefficients) are obtained with the compounds of highest aqueous solubility. This latter relation is illustrated in Table II, the fluorescent brightening agent (X), which has a different chemical constitution, being anomalous.

TABLE II

Dye	Aqueous Solubility at 60°C (mg/litre)	Partition Coefficient at 60°C	Fibre Saturation Value at 60°C (calc.) (g/100 g dry viscose rayon)
V	3.0 <sup>a</sup>	(32.7)	0.010
VI	3.8 <sup>a</sup>	19.3	0.008
IX	7.8 <sup>a</sup>	(41.4)	0.032
VIII	7.9	59.7	0.047
VII <sup>a</sup>	12.3	27.0	0.033
VII	12.3	32.5	0.040
X	31.5	70.6	0.220
III	74	10.5	0.078
I	141 <sup>a</sup>	9.1	0.114
II	214 <sup>a</sup>	6.0	0.128
XII	404	7.3	0.205
IV	460	7.0	0.320
XIII	1370	1.8	0.245
XI	1510	4.7	0.710

Values in parentheses were obtained by extrapolation.

<sup>a</sup> On cotton.

With the seven compounds whose affinities were determined at three temperatures, a linear relation was observed when  $-\Delta\mu^\circ/T$  was plotted against  $1/T$ , and it was assumed that the other six compounds would behave similarly. Where a comparison is possible, the heats of dyeing are found to resemble those of wool rather than those of cellulose acetate fibre. On cellulose acetate<sup>5</sup>, heats of dyeing are fairly constant at approx. 10 kcal/mole, whereas on cellulose and wool they show considerable variation and are always lower. It seems probable that non-ionic dyes are attracted to cellulosic fibres through non-polar van der Waals forces.

Table I shows a wide spread of values for the entropy change. The average value (–12 cal/mole per deg. C) is higher than that for wool<sup>1</sup>

(-8.5), but lower and much less constant than for cellulose acetate<sup>5</sup> (-17), and much lower than for direct dyes on cellulose<sup>4</sup> (-35).

A statistically significant inverse correlation was found between  $-\Delta H^\circ_{\text{dye}}$  and the logarithm of the aqueous solubility at 60°C, and a direct correlation between  $-\Delta H^\circ_{\text{dye}}$  and  $\Delta H^\circ_{\text{H}_2\text{O}}$ .

The latter correlation follows from equation (4), since an increase in  $\Delta H^\circ_{\text{H}_2\text{O}}$  will increase the (negative) value of  $\Delta H^\circ_{\text{dye}}$ , if  $\Delta H^\circ_{\text{fibre}}$  is constant. Table I shows that, with two exceptions,  $\Delta H^\circ_{\text{fibre}}$  is reasonably constant.

A possible explanation of the inverse correlation between heat of dyeing and aqueous solubility lies in the suggestion that combination between non-ionic dyes and cellulose takes place through non-polar van der Waals forces. Heat of dyeing is a measure of the strength of these forces, and increases as the dye becomes less hydrophilic, e.g. owing to the presence of an additional benzene ring (cf. II and IX).

The usual relation was found between the heat of dyeing and the entropy change, expressed by the equation—

$$\Delta H^\circ_{\text{dye}} = 0.475 \Delta S^\circ + 0.62$$

Two of the dyes (II and IV) were sufficiently

soluble at room temperature to enable spectrophotometric curves to be obtained (using 0.1, 1.0, and 4.0-cm cells) over the concentration ranges 0–19 and 0–42.5 mg/litre, respectively. There was no sign of any change in the shape of the curves as the concentration of dye increased, so it is concluded that non-ionic dyes are not aggregated in aqueous solution.

\* \* \*

We wish to thank Courtaulds' Scientific and Educational Trust Fund for a scholarship which enabled one of us (J.R.A.) to take part in this work.

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING  
THE UNIVERSITY  
LEEDS 2

(Received 31st August 1960)

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## The Dyeing of Cellulose Acetate with Disperse Dyes X—Saturation Values with Mixtures of Dyes

C. L. BIRD and P. RHYNER

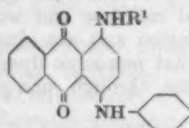
With disperse dyes which are insufficiently soluble in cellulose acetate fibre to yield a full shade, the difficulty can be overcome by using a mixture of two or three dyes of similar hue and chemical composition. The components of the mixture must be carefully chosen, as some dyes interact to give a compound which has low solubility in the fibre. There is no evidence that the solubility in cellulose acetate of a mixture of disperse dyes can exceed the sum of the solubilities of the individual components. Interaction can also occur when a mixture of disperse dyes is dissolved at 22°C in ethanol, hexane, or 0.2% aqueous Ultravon W.

### INTRODUCTION

In general, the behaviour of secondary cellulose acetate towards disperse dyes is that of a solvent. At any given temperature, the fibre is capable of dissolving a definite amount of dye. The maximum solubility, or fibre saturation value, varies from one dye to another, and increases with increasing temperature.

If more than one dye is present, the dyes usually dissolve in the fibre independently, i.e. without interaction, but in some cases, with dyes of similar chemical constitution, interaction does occur, and the total solubility may be less than that of one of the individual components of the mixture.

Vickerstaff and Waters<sup>1</sup> examined four dyes of general formula—



where R<sup>1</sup> was methyl (I),  $\beta$ -hydroxyethyl (II), isobutyl (III), and ethyl (IV). With binary

mixtures of these dyes, interaction occurred on cellulose acetate at 85°C when IV was present with I or II, but not when it was mixed with III. No interaction occurred with the mixtures (I + II) and (I + III). Interaction was considered to have taken place when the fibre saturation value of the mixture was less than that of one of the individual components.

Another case of interaction has been recorded by Schuler and Remington<sup>2</sup>, who found that, on polyethylene terephthalate at 89.3°C, the saturation values of 1-amino-4-hydroxyanthraquinone and 1,4-dihydroxyanthraquinone were decreased by about 20% when one of the dyes was applied in presence of a large amount of the other.

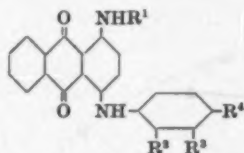
Saturation values are important in practice, because some of the blue anthraquinonoid dyes, e.g. I, have low saturation values and will not "build-up" to give deep dyeings. This difficulty can be overcome by using a mixture of dyes, if the saturation values are additive. Thus, Ciba Ltd have patented<sup>3</sup> the use of a mixture of I and II. They point out that this mixture, besides giving deep dyeings, gives blues of great purity. This latter point is important, since mixing of dyes is liable to result in a dull hue. The use of such

additive mixtures was first patented by Ciba Ltd in 1932<sup>4</sup>.

Sandoz Ltd<sup>5</sup> claim that dyes of the 1-amino-4-phenylaminoanthraquinone series which have little affinity for cellulose acetate show greatly increased affinity in the form of mixtures. The Sandoz patent implies that such a mixture would produce a deeper dyeing than that obtainable if each of the individual dyes was taken up independently. No evidence, however, has been published which supports this view.

#### OUTLINE OF THE INVESTIGATION

The work of Vickerstaff and Waters<sup>1</sup> has been repeated, and this work has been extended by examining a similar series of dyes of general formula—



where R¹, R², R³ and R⁴ are—

Dye No.	R¹	R²	R³	R⁴
I	CH₃	H	H	H
V	CH₃	H	H	CH₃
VI	CH₃	CH₃	H	H
VII	CH₃	H	CH₃	H
VIII	CH₃	CH₃	H	CH₃

In addition, the two dyes found by Schuler and Remington<sup>2</sup> to interact on polyethylene terephthalate, viz. 1,4-dihydroxy-(IX) and 1-amino-4-hydroxy-anthraquinone (X), have been examined on cellulose acetate.

The solubility of dyes I-VIII has been determined at 22°C in absolute alcohol, hexane, and 0.2% aqueous Ultravon W (Ciba). Times of half-dyeing have been determined for dyes I-VIII.

#### EXPERIMENTAL

All dyeings were carried out with 1-g hanks of scoured cellulose acetate at 85°C in a Marney machine. The purified dyes were in the form of fine dispersions provided by Ciba Ltd. These dispersions, which had a particle size of 2–3  $\mu$ , were prepared by ball-milling 1 part of dye with 1 part of Ultravon W and 8 parts of water. (With dyes of the type investigated, very fine dispersion is

essential.) As all the dyes were slow-dyeing, 24 h were allowed for equilibrium to be established. Ten mg of each dye were used with the blue anthraquinonoid dyes (I-VIII), e.g. with a binary mixture (10 + 10) mg of dye were used; with IX and X 20 mg of each dye were used. The liquor:yarn ratio was 100:1, and 2 g of Ultravon W (a derivative of sodium  $\mu$ -heptadecylbenzimidazole disulphonate<sup>6</sup>) were added per litre of dyebath. After being dyed, the yarn was rinsed well and dried in a room conditioned at 65% r.h. and 22°C. A portion of the yarn (0.1 g) was then dissolved in acetone, and the dye present was determined by colorimetric analysis in a Unicam SP600 spectrophotometer.

All the blue anthraquinonoid dyes (I-VIII) had similar absorption curves, with peaks at 600 and 640 m $\mu$ . Dye on the fibre was measured at 590 m $\mu$  (Vickerstaff and Waters used 595 m $\mu$ ). With mixtures, a calibration curve was prepared from a mixture of equal weights of the individual components. Although the dyes were not necessarily taken up in these proportions by the fibre, the error introduced was sufficiently small to be ignored.

TABLE I

Dye	Molecular Weight	Melting Point (°C)	Molar Extinction Coefficient (e) in Acetone at	
			600 m $\mu$	640 m $\mu$
I	328	140–141	16,700	17,100
II	358	208–209	15,200	16,100
III	370	111–112	15,700	15,700
IV	342	181–182	15,800	16,300
V	342	182–183	14,700	15,700
VI	342	128–129	12,000	12,700
VII	342	112–113	13,700	14,400
VIII	356	180–181	15,600	17,200

With IX and X, measurements were made at the peak wavelengths, and the amount of each dye present in mixtures was calculated by means of the formulae given by Vickerstaff<sup>7</sup>.

The solubilities of crystalline samples of the dyes were determined at 22°C in absolute alcohol and in hexane, using 100 mg of each dye and 10 ml of solvent. Ten days (with frequent shaking) were allowed for equilibrium to be attained. The solution was then filtered and the concentration of dye determined spectrophotometrically. Table I gives the molecular weights, melting points and molar extinction coefficients of

TABLE II

Dye	Solubility (mg/litre) at 22°C in				Fibre Saturation Value at 85°C (mg/g)	Time of Half-dyeing at 85°C (min)
	Ethanol	Hexane	0.1% Aqueous Ultravon W	(a/b)		
	(a)	(b)	(c)			
I	550	270	9.3	2.0	6.1	41
II	350	0	19.1	—	4.1	32
III	3130	3000	10.5	1.0	5.1	100
IV	130	65	0.75	2.0	1.4	30
V	450	130	6.0	3.5	2.0	25
VI	2030	1290	26.1	2.1	6.0	46
VII	1650	910	16.8	1.8	7.2	42
VIII	230	110	7.2	2.1	1.2	20



TABLE III  
Fibre Saturation Values of Mixtures at 85°C

Mixture		Fibre Saturation Value (mg/g)		Difference (%)
		Calculated	Found	
I + II	III	10.2	10.3	+1
	IV	11.2	11.4	+2
	V	7.5	4.7	-37
	VI	8.1	8.7	+7
	VII	12.1	11.3	-7
	VIII	13.3	13.6	+2
	IX	7.3	7.8	+7
	X	9.2	8.0	-13
II + III	IV	5.5	3.5	-36
	V	6.1	5.9	-3
	VI	10.1	10.4	+3
	VII	11.3	12.0	+6
	VIII	5.3	5.3	0
	IX	6.5	5.4	-17
	X	7.1	7.1	0
	XI	11.1	10.4	-6
III + IV	V	12.3	11.0	-11
	VI	6.3	6.1	-3
	VII	3.4	3.4	0
	VIII	7.4	4.8	-35
	IX	8.6	8.7	+1
	X	2.6	2.6	0
	XI	8.0	6.2	-23
	XII	9.2	9.6	+4
V + VI	VII	3.2	2.9	-9
	VIII	13.2	9.6	-27
	IX	7.2	4.2	-42
	X	8.4	7.7	-8
	XI	14.1	12.5	-11
	XII	15.3	15.5	+1
	XIII	19.3	19.5	+1
	XIV	15.2	11.4	-25

the dyes, and Table II the solubilities in the two liquid solvents, as well as the fibre saturation values and times of half-dyeing. The fibre saturation values are in units of mg dye per g of air-dry fibre.

The solubilities in 0.2% aqueous Ultravon W, also given in Table II, were determined by stirring 50 mg of the crystalline dye in 50 ml of the solution at 85°C for 4 h in the Marney machine, cooling, leaving the solution for 3 days at 22°C, removing a portion of the solution through a filter, and estimating the dye present as before.

Rate-of-dyeing curves were obtained by carrying out dyeings with fine dispersions of purified dyes as for the determination of saturation values, but for different times. The time of half-dyeing ( $t_{0.5}$ ) is the time required to reach half the exhaustion obtained after 24 h. The values given in Table II are probably dependent as much on particle size (which governs rate of dissolution) as on rate of diffusion in the fibre.

## DISCUSSION

## Saturation Values

The measured values for dye on the fibre given in Tables II and III are described as "saturation values". This is not quite correct for VI and VII, where the true fibre saturation values are

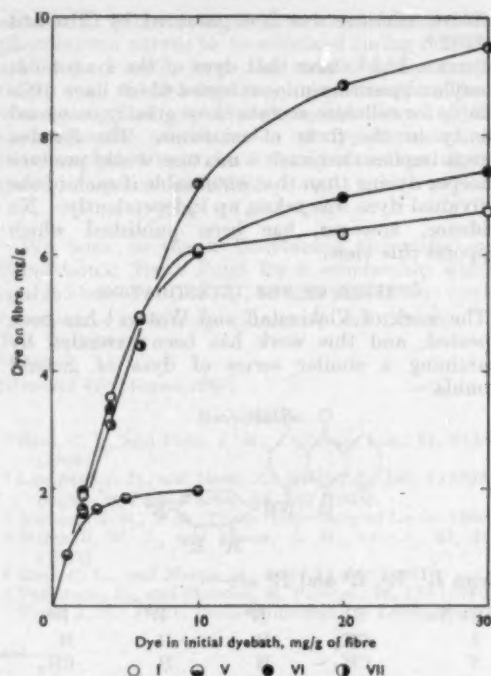


FIG. 1.—Adsorption isotherms at 85°C

7.0–7.5 mg and 9.0–9.5 mg per g, respectively (see Fig. 1), but to attain these values, considerably more than 10 mg of each dye would have been required. With anthraquinonoid disperse dyes, there is a probability of surface adsorption in presence of a large excess of dye, and this would explain the gradual slope of the second part of the curves given by Vickerstaff and Waters<sup>1</sup>. (This effect is also seen with I in Fig. 1.) With the other dyes used, the values given are believed to approximate to true saturation values. It was considered undesirable in the work to use a large excess of dye, and the conclusions are not dependent on complete saturation of the fibre.

The results given in Table III for binary mixtures are summarised in Table IV, where i = interaction and ni = no interaction.

TABLE IV  
Interaction in Binary Mixtures of Dyes

	II	III	IV	V	VI	VII	VIII
I	ni	ni	i	ni	ni	ni	ni
II		ni	i	ni	ni	ni	ni
III			ni?	ni	ni	ni	ni
IV				ni	i	ni	ni
V					i?	ni	ni
VI						i?	i
VII							ni

The percentage difference between the sum of the individual fibre saturation values and the saturation value of the mixture shows a range of +7 to -42%. The positive values may be due to experimental error. With the mixtures (IV + I), (IV + II), (VI + IV), and (VI + VII), the difference exceeds -34% and the saturation value of the mixture is less than that of one of the individual



components. The mixtures (IV + III), (VI + V), and (VI + VII) are regarded as borderline cases. Where interaction occurs it is always associated with the presence of either IV or VI, but IV does not interact with V, VII and VIII, whilst VI does not interact with I, II and III. The interaction found with IV confirms the results of Vickerstaff and Waters<sup>1</sup>, and, like these authors, we are unable to explain why interaction occurs in some cases and not in others.

Fig. 2 illustrates the build-up of a binary mixture where no interaction takes place, whilst Fig. 3 shows the effect of interaction.

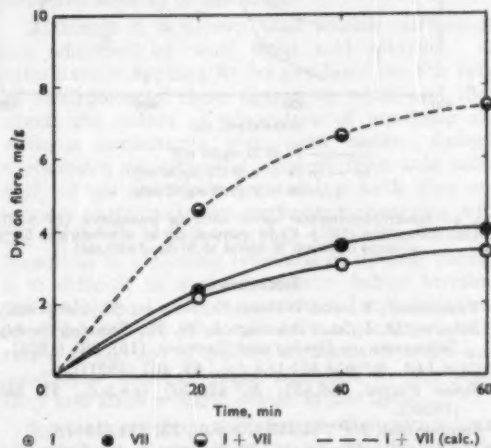


Fig. 2—Rate curves at 85°C for dyes showing no interaction

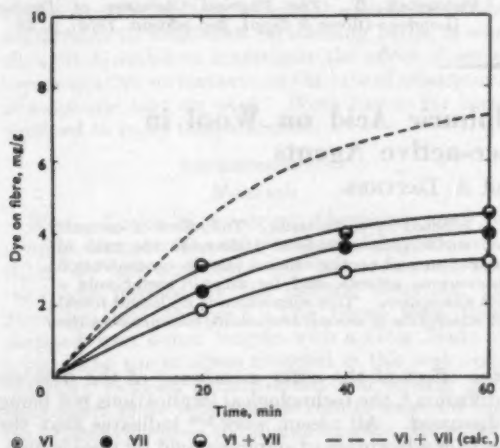


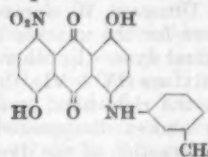
Fig. 3—Rate curves at 85°C for dyes showing interaction

The saturation values of the individual dyes (Table II) range from 0.12% to 0.72%. It is clear that the dyes with the lowest saturation values (IV, V and VIII) are of no commercial interest. None of the dyes alone will build up to a really deep blue, but much deeper dyeings can be obtained by using a mixture of two or three of the dyes. The best result is obtained with a mixture of I, VI and VII, which gives a fibre saturation value of nearly 2%. This mixture, however, would probably have an undesirably low melting point

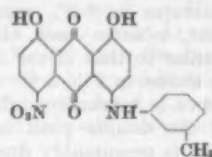
(see Table I). It is noteworthy that, apart from II (where the hydroxyl group probably produces intermolecular hydrogen bonding in the crystal), the dyes with the highest solubility in the fibre have the lowest melting points.

Results for the two dyes (IX and X) used by Schuler and Remington showed a fall of saturation value of about 20%, so that these dyes behave similarly on cellulose acetate and on polyethylene terephthalate.

At one stage in the investigation it was thought that an example had been found where the saturation value of a binary mixture exceeded the sum of the saturation values of the individual components. The two dyes—



(XI)



(XII)

were examined, as well as a mixture of the two dyes (XIII) prepared by reaction of a mixture of the intermediates for XI and XII with *m*-toluidine.

The saturation value of XIII was found to be more than twice the sum of the saturation values of XI and XII. However, further investigation (which included chromatographic separations) showed that the saturation values found for XI and XII were too low, owing to the use of insufficiently fine dispersions, the correct values being 1.58 and 1.31 mg/g of fibre, respectively, whilst the value for XIII was 2.13 mg/g.

#### Interaction in Solution

In Table V are given solubilities at 22°C of mixtures of dyes in 0.2% aqueous Ultravon W, and in Table VI are given solubilities in absolute alcohol and in hexane. The only correlation observed is between the solubilities in absolute alcohol and in hexane, except with II, where the hydroxyl group renders the dye insoluble in hexane. It is also noteworthy that dyes with the highest solubility in cellulose acetate at 85°C are the most soluble in 0.2% aqueous Ultravon W at 22°C.

TABLE V  
Solubilities of Dye Mixtures at 22°C in 0.2% Aqueous Ultravon W

Mixture	Solubility (mg/litre)		Difference (%)	
	Calculated	Found	Ultravon Solution	On Fibre (from Table III)
I + III	19.8	15.8	-20	+2
IV	10.0	7.3	-27	-37
VI	35.3	24.3	-31	-7
II + IV	19.8	11.2	-43	-36
VI	45.1	35.5	-21	+3
IV + V	6.8	7.0	+3	0
VI	26.8	16.3	-39	-35
VII	17.5	20.8	+19	+1
VIII	8.0	6.5	-19	0
V + VI	32.0	18.3	-43	-23
VI + VII	42.8	25.8	-40	-27
VIII	33.2	18.0	-46	-42

TABLE VI  
Solubilities of Dye Mixtures at 22°C in Absolute Alcohol and in Hexane

Mixture	Solubility (mg/100 ml)		Difference (%)	
	Absolute Alcohol	Hexane	Absolute Alcohol	Hexane
	Cale.	Found	Cale.	Found
I + IV	68	65	33	32
VI	318	250	154	102
IV + V	58	50	19	21
VII	178	162	97	107
V + VI	308	281	140	122
VI + VII	428	306	218	127

Spectrophotometric curves of solutions of dye mixtures in 0.2% aqueous Ultravon W showed that, in some cases, the curve for the mixture is similar to that of the individual dyes. In others, as shown in Fig. 4 for the mixture (IV + VI), the curve is markedly different, the right-hand peak of the double peak having almost disappeared. This is presumably due to interaction of the dyes in solution, but there is no correlation with interaction in cellulose acetate. Table V includes one case (IV + VII) where the solubility of the mixture exceeds the sum of the solubilities of the individual dyes.

The authors thank Ciba Ltd for sponsoring this work, and for giving leave of absence to one of us (P.R.) to enable him to take part in the investigation.

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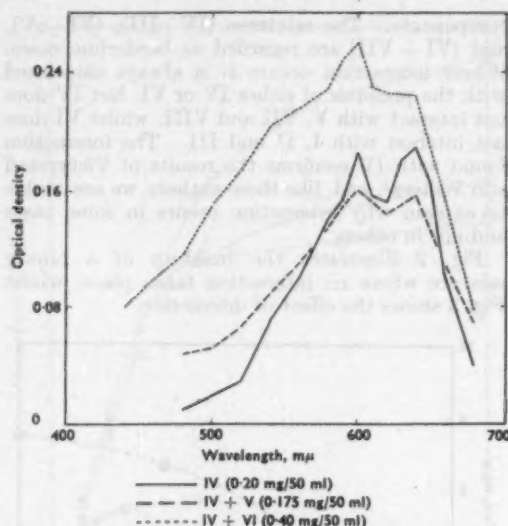


FIG. 4—Spectrophotometric curves showing interaction (IV + VI) and no interaction (IV + V) in solution (20 ml of solution in 0.2% aqueous Ultravon W added to 30 ml of acetone)

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## Rates of Adsorption of Sulphuric Acid on Wool in Presence of Surface-active Agents

N. BUCHSBAUM and A. DATYNER

The rate of adsorption of sulphuric acid on wool is affected by surfactants. The effect of cationic and non-ionic surfactants is small, but some anionic surfactants considerably decrease the rate of adsorption of acid, the effect depending on the concentration and on the critical micelle concentration of the agent. All agents are themselves adsorbed to various extents, and for anionic surfactants a correlation appears to exist between acid and surfactant adsorption. This correlation is no longer found if sodium sulphate is added, since this reduces the rate of adsorption of anionic surfactant, but increases the rate of adsorption of acid.

#### INTRODUCTION

The adsorption of acids on wool and other animal fibres under equilibrium conditions has received considerable study<sup>1</sup>. These investigations were concerned with the elucidation of the mechanism of attachment between acids and the fibre, and served mainly to explain the mechanism of dyeing with levelling acid dyes and the function of acid and Glauber's salt (sodium sulphate) in this process. Other investigators have dealt with the degradation of wool when acids are used in such processes as dyeing or carbonising<sup>2</sup>.

In contrast, little attention has been paid to rates of adsorption of acid by wool. Even then, work was concerned almost exclusively with the mechanism of diffusion of acids into wool<sup>3</sup> and

the effect of the outer membrane of the fibre on diffusion<sup>4</sup>, the technological implications not being discussed. All recent work<sup>3-6</sup> indicates that the rate of adsorption of sulphuric acid by wool is high.

High rates of adsorption may lead to uneven distribution of acid in assemblies of wool fibres, because it is impossible to effect simultaneous intimate contact between the bath and all the fibres. Once hydrogen ions have been adsorbed unevenly, their redistribution may be difficult. This is why, in processes where acids are applied to wool, the fibres are brought into contact with the acid bath at as low a temperature as is practical, e.g. room temperature for carbonising and 50–60°C for dyeing. The higher temperature in the latter case facilitates the dyeing process because the

fibre is then more swollen and is more easily wetted. Any unevenness which does occur, probably as a result of uneven acid distribution, is remedied by subsequent boiling. Although boiling is necessary for other reasons, there is little doubt that, if uneven dye distribution results from uneven initial adsorption, the time required for complete levelling is unduly prolonged.

In recent years great interest has been shown in the use of surface-active agents (surfactants) in various dyeing and finishing processes. These agents can promote levelling in several ways, one of their functions being to assist in quick and effective wetting of the fibre.

Although it is known that anionic surfactants are adsorbed by wool from acid solution<sup>7</sup>, no information appears to be available on the rates of adsorption of these agents on wool, and little about the extent of adsorption of non-ionic and cationic surfactants from acid baths. Anionic surfactants are adsorbed strongly from acid baths and, in an acid bath containing both dye and anionic surfactant, the surfactant competes with the dye. It is believed that this competition is beneficial in obtaining levelness of dyeing, though it is difficult to understand how better levelling can be achieved unless uneconomical concentrations of surfactant are used. In fact, objections can be raised to the use of anionic surfactants under acid conditions because, being adsorbed by the wool, they lose their wetting action in the bath.

Since chemical manufacturers often recommend the addition of surfactants to practically all baths used in wool dyeing and finishing, and because of the discovery of the beneficial effects of some surfactants in loose-wool carbonising baths, it was thought desirable to investigate the effect of some representative surfactants on the rate of adsorption of sulphuric acid on wool. Work has so far been confined to room temperatures.

#### EXPERIMENTAL

##### Materials

**Wool**—Locks of pen-grown Corriedale wool of average diameter  $30\ \mu$  were used for all experiments. After the tips and roots had been removed, the wool was purified by the method described by Swanston and Palmer<sup>8</sup>. The fibres were then chopped into 4-mm lengths with a razor blade in a jig. The use of fibres chopped in this way considerably reduced entanglement and felting, and gave much more reproducible results than did the use of fibres chopped to random lengths. However, it should be noted that with short fibres diffusion through fibre ends probably becomes significant. It was also found that handling of wool after purification affected adsorption; rubber gloves were therefore used. Fibres were conditioned and weighed in a room maintained at  $70^{\circ}\text{F}$  ( $21^{\circ}\text{C}$ ) and 65% r.h.

**Surface-active Agents**—Samples of pure sodium alkyl sulphates, sodium dialkyl sulphosuccinates, alkyltrimethylammonium bromides, and alkylpyridinium chlorides were used without further purification. A technical grade of sodium dodecylbenzenesulphonate was extracted with butan-1-ol

from an aqueous solution in a Kieselbach extractor<sup>9</sup>. The solvent was distilled at reduced pressure and the residue recrystallised three times from petroleum ether ( $60\text{--}80^{\circ}\text{C}$ ).

Two of the alkylaryl polyethylene oxide non-ionic surfactants were octyltolyl and two were nonylphenyl polyethylene oxides, varying in ethylene oxide chain length. Two other non-ionic surfactants were condensates of ethylene oxide and propylene oxide of average molecular weight 3000 and 8000, prepared from 40–50% and 80–90% ethylene oxide and 60–50% and 20–10% propylene oxide, respectively. These agents were used without further purification.

**Reagents**—Sulphuric acid and other inorganic chemicals were of analytical reagent quality. Rosaniline hydrochloride (C.I. Basic Violet 14) was obtained pure. A sample of Naphthalene Orange G (C.I. Acid Orange 7) was purified by crystallisation from ethanol. These dyes were used for the determination of anionic and cationic surfactants, respectively<sup>10,11</sup>.

##### Apparatus

Experiments were carried out in 100-ml conical flasks provided with ground-glass stoppers, immersed to one-third of their height in a thermostat at  $25^{\circ}\text{C}$ . The flasks were fixed to a laboratory shaker by means of a special attachment which produced a sideways movement instead of the usual up-and-down oscillation. The latter was unsuitable because it caused splashing, prevented continuous immersion of the flasks in the thermostat bath to a sufficient depth, and made the withdrawal of solution samples difficult.

##### Procedure

Samples of conditioned wool (1 g) were wetted out in distilled water at  $50^{\circ}\text{C}$ . Excess of water was removed by means of suction and the damp fibre pads were introduced into the flasks held in the shaker. Wool pre-wetted in this way contained consistently 75% of its own weight of water. Pre-wetting by a standard method was found to be essential to obtain reproducibility.

Solutions of acid, surfactants, or mixtures of both of appropriate concentration were also kept in flasks immersed in the thermostat. Reagent solution (50 ml) was added quickly with shaking. At the required times, 1–20-ml samples were withdrawn through a glass-wool plug attached to the tip of a pipette, and the acid content was determined. It was originally thought that it might be possible to follow the adsorption of sulphuric acid by wool by means of continuous pH measurement with a glass electrode. This was satisfactory when only sulphuric acid was used, but when surfactants were present deviations from the "true" pH of the solutions were found, so that this method of measuring acid concentration had to be abandoned. Thus, the pH of a 0.01-N solution of sulphuric acid was 2.18, but in presence of sodium dodecylbenzenesulphonate ( $10^{-3}\text{M}$ ) the pH was 2.08.

Potentiometric titration was tried as an alternative method, but it was too slow and no more accurate than titration with 0.008-N caustic



soda from a microburette, using phenolphthalein as indicator. Surfactants had no effect on the end-point of the titration.

Cationic and anionic surfactants were determined by methods described by Few and Ottewill<sup>10</sup> and by Karush and Sonenberg<sup>11</sup>, respectively. Colorimetric measurements were carried out on a Beckman DK2 Recording Spectrophotometer, which was also used for determination of alkylaryl polyethylene oxide non-ionic surfactants by ultraviolet absorption spectra<sup>12</sup>.

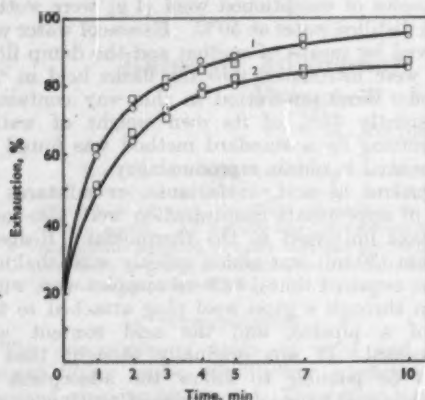
Shaking wool with anionic and with some non-ionic surfactants produced a cloudiness in the bath, the intensity of which increased on standing. The nature of this cloudiness has not been investigated, but because of it, samples had to be centrifuged before spectrophotometric measurements were made. To reduce variability due to adsorption of surfactants on glass surfaces, all manipulations were carefully standardised.

Critical micelle concentrations were determined by means of surface tension measurements on a du Noüy tensiometer.

## RESULTS

### Sulphuric Acid in Absence of Surfactants

The amount of sulphuric acid adsorbed at different times (expressed as percentage exhaustion of the sulphuric acid originally present in the bath) is shown in Fig. 1. Each result is the mean of at least two independent determinations. About 80–90% of sulphuric acid (depending on the original concentration) is adsorbed in the first five minutes, the proportion being higher at the lower concentration. The rate of adsorption of acid was not affected by sodium sulphate.



- 1 ○ 0.005-N  $H_2SO_4$   
 □ 0.005-N  $H_2SO_4$  + 2 g/l. sodium sulphate  
 2 □ 0.01-N  $H_2SO_4$   
 □ 0.01-N  $H_2SO_4$  + 2 g/l. sodium sulphate

FIG. 1—Adsorption of sulphuric acid

### Sulphuric Acid in Presence of Surfactants

The addition of surfactants affects the rate of adsorption of sulphuric acid (Fig. 2). Both alkylaryl polyethylene oxide non-ionic surfactants and cationic surfactants slightly accelerate adsorption, whereas anionic surfactants reduce the rate.

Condensates of ethylene oxide and propylene oxide appear to have no effect.

Since it was desired to examine anionic surfactants in greater detail, attention was focused on two alkyl sulphates, three dialkyl sulphosuccinates and sodium dodecylbenzenesulphonate. Almost all of these are freely available commercially, though some only in technical grades. The concentrations of surfactants used were approximately those recommended for industrial textile processes.

At equivalent concentrations the effect of different anionic surfactants on acid adsorption increases with molecular weight within each series (Fig. 3), but no correlation exists between different series. Sodium dodecylbenzenesulphonate (mol. wt. 349) has a similar effect to that of sodium bis-tridecyl sulphosuccinate (mol. wt. 584), whereas

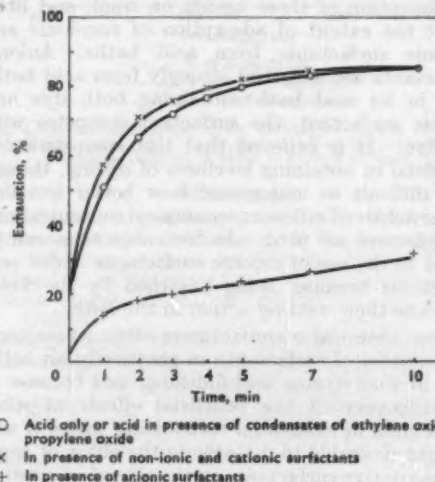


FIG. 2—Adsorption of sulphuric acid from a 0.01-N solution in presence of surfactants

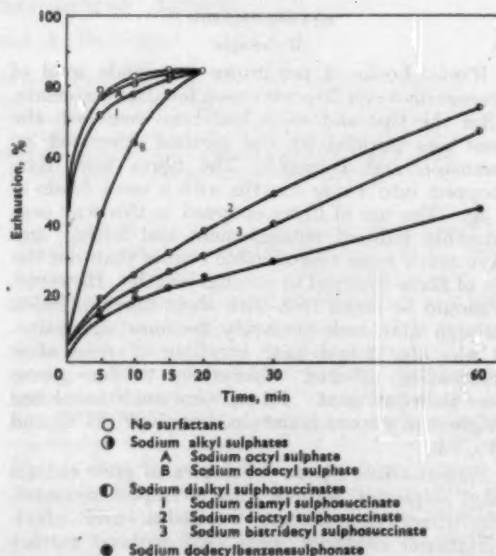


FIG. 3—Adsorption of sulphuric acid from a 0.01-N solution in presence of anionic surfactants ( $10^{-3}$  M)



sodium dodecyl sulphate (mol. wt. 288) is more effective than sodium diamyl sulphosuccinate (mol. wt. 360). It was not possible to compare the effects of sodium hexadecyl sulphate (mol. wt. 344) and sodium dodecylbenzenesulphonate (mol. wt. 348), because the former was not sufficiently soluble at 25°C.

It seems that the effectiveness of anionic surfactants in reducing the rate of adsorption of sulphuric acid is related to the existence of micelles in solution, since the surfactants with lower critical micelle concentrations were found to be the more effective. The critical micelle concentrations of the surfactants are given in Table I.

TABLE I  
Critical Micelle Concentration (cmc) of some Anionic Surfactants

Surfactant	cmc (millimoles per litre)	Acid absent	0.01-N H <sub>2</sub> SO <sub>4</sub>
Sodium dodecyl sulphate (C <sub>12</sub> )	6.3*	—	4.0*
Sodium dodecylbenzenesulphonate (DDB)	1.0*	—	0.6*
Sodium diamyl sulphosuccinate (AM)	27.0†	—	—
Sodium dioctyl sulphosuccinate (OC)	1.6†	—	—
Sodium bistridecyl sulphosuccinate (TR)	0.5†	—	—

\* Obtained from surface tension measurements.

† Reference 13.

The concentration of surfactant at which the experiments were carried out (one millimole per litre) is above the critical micelle concentration of the two surfactants (TR and DDB) which have the greatest effect in reducing the rate of adsorption of acid.

It was expected that the presence of acid would reduce the critical micelle concentration of anionic surfactants, in accordance with the effect of gegenions on surfactants<sup>14,15</sup>. This was borne out experimentally, as shown in Table I. Thus the critical micelle concentration of DDB was reduced

in presence of 0.01-N sulphuric acid to below one millimole per litre. An increase in the concentration of surfactant decreases further the rate of adsorption of acid, but with a surfactant with a high critical micelle concentration (such as C<sub>12</sub>), a concentration as high as  $6 \times 10^{-3}$  M has little more effect than has  $4 \times 10^{-4}$  M DDB (Fig. 4).

#### Surfactants in Presence of Sulphuric Acid

Results shown in Table II indicate that, in the absence of sulphuric acid, cationic surfactants are adsorbed considerably, the amount adsorbed increasing with increasing molecular weight within a series. On the other hand, anionic surfactants are adsorbed only slightly, and again the amount adsorbed increases with increasing molecular weight (Table III). Alkylaryl polyethylene oxide non-ionic surfactants show an intermediate behaviour, the extent of adsorption being inversely related to the length of the polyethylene oxide chains. These results confirm observations by Rose, Weatherburn, and Bayley<sup>16</sup>, but are contrary to those of McLaren<sup>17</sup>, who found no adsorption of an unspecified non-ionic surfactant whose detergency properties he examined. It is possible that the polyethylene oxide chain was of such a length as to render adsorption very small and not measurable by the method used.

Addition of sulphuric acid causes considerable reduction in the adsorption of cationic surfactants and a considerable increase in the adsorption of anionic surfactants, whilst the adsorption of alkylaryl polyethylene oxide non-ionic surfactants is increased only slightly. McLaren found that his non-ionic agent was adsorbed from acid solution. Since the rates of adsorption of cationic and non-ionic surfactants in presence of acid are small, the full curves are not presented. A comparison of the amounts of the various types of surfactants adsorbed on wool after 60 min, in absence and presence of 0.01-N sulphuric acid, is given in Tables II, III and IV.

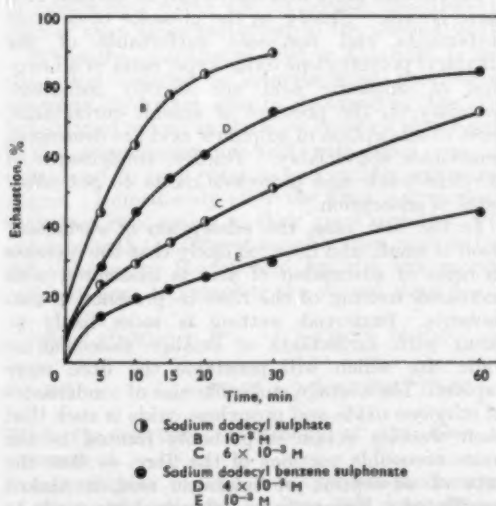


Fig. 4—Adsorption of sulphuric acid from a 0.01-N solution in presence of sodium dodecyl sulphate and sodium dodecylbenzenesulphonate at concentrations below and above the cmc

TABLE II  
Percentage Exhaustion of Cationic Surfactants on Wool after 60 min

Surfactant (Original concn. $10^{-3}$ M)	Percentage exhaustion Acid absent	0.01-N H <sub>2</sub> SO <sub>4</sub>
Decyltrimmonium bromide	32.8	3.7
Dodecyltrimmonium bromide	44.2	4.2
Decylpyridinium chloride	55.8	8.1
Tetradecylpyridinium chloride	58.3	20.1

TABLE III  
Percentage Exhaustion of Anionic Surfactants on Wool after 60 min

Surfactant (Original concn. $10^{-3}$ M)	Percentage exhaustion Acid absent	0.01-N H <sub>2</sub> SO <sub>4</sub>
Sodium octyl sulphate	11.3	—
Sodium dodecyl sulphate	17.1	99.0
Sodium diamyl sulphosuccinate	1.9	—
Sodium dioctyl sulphosuccinate	8.1	58.7
Sodium bistridecyl sulphosuccinate	8.6	—
Sodium dodecylbenzenesulphonate	—	27.0

TABLE IV  
Percentage Exhaustion of Non-Ionic Surfactants on Wool after 60 min

Surfactant (Original concn. $5 \times 10^{-4}$ M)	Percentage exhaustion	
	Acid absent	0.01-N $H_2SO_4$
Octyltolyl polyethylene oxide (16)*	7.7	9.0
Octyltolyl polyethylene oxide (9.5)*	11.3	10.0†
Nonylphenyl polyethylene oxide (15)*	8.7	11.3
Nonylphenyl polyethylene oxide (9)*	10.2	16.8

\* Average number of ethylene oxide links.

† Difficulties were experienced with ultraviolet measurements owing to the low solubility of this surfactant.

Rates of adsorption of anionic surfactants in presence of acid are shown in Fig. 5, which compares the different anionic surfactants at equimolar

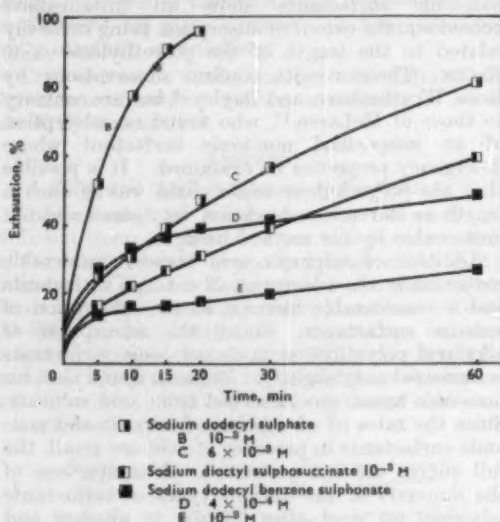


FIG. 5—Adsorption of anionic surfactants in presence of 0.01-N sulphuric acid (original concentration)

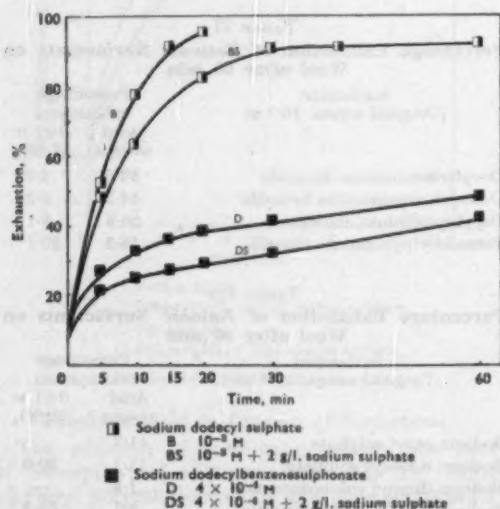


FIG. 6—Adsorption of sodium dodecyl sulphate and sodium dodecylbenzenesulphonate from solutions containing 0.01-N sulphuric acid and 2 g/l. sodium sulphate

concentrations and shows the effect of concentration on adsorption.

#### Anionic Surfactants in Presence of Sulphuric Acid and Sodium Sulphate

The amount of anionic surfactant removed from solution by wool in the presence of sulphuric acid is considerable. It was thought that the presence of sodium sulphate, since it lowers the critical micelle concentration of anionic surfactants<sup>18</sup>, might reduce the rate of adsorption of the surfactant. Fig. 6 shows this to be the case. However, the effect of the surfactants on the rate of adsorption of sulphuric acid is almost eliminated (Fig. 7).

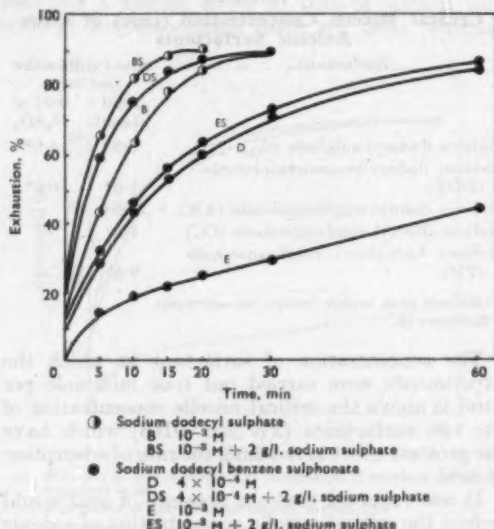


FIG. 7—Adsorption of sulphuric acid from 0.01-N solution containing anionic surfactants and 2 g/l. sodium sulphate

#### DISCUSSION

The results presented above can be divided into three groups. Firstly, in the presence of cationic surfactants and non-ionic surfactants of the alkylaryl polyethylene oxide type, rates of adsorption of sulphuric acid are slightly increased. Secondly, in the presence of anionic surfactants, rates of adsorption of sulphuric acid are decreased, sometimes appreciably. Thirdly, condensates of ethylene oxide and propylene oxide do not affect rates of adsorption.

In the first case, the adsorption of surfactant itself is small, and it seems likely that the increase in rates of adsorption of acid is associated with increased wetting of the fibre in presence of surfactants. Improved wetting is more likely to occur with surfactants of smaller molecular or ionic size which will penetrate the fibre more rapidly. The average molecular size of condensates of ethylene oxide and propylene oxide is such that their wetting action is probably limited to the more accessible portions of the fibre, so that the rate of adsorption of sulphuric acid is almost unaffected. Reference has already been made to the importance of using a standard method of pre-wetting fibres; it seems that submerging wool fibres

in aqueous solutions until they no longer return to the surface provides no guarantee that air has been completely displaced from the interior of the fibre.

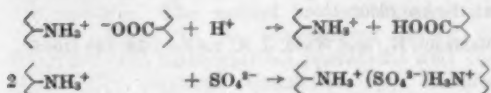
The surface potential of wool immersed in neutral solutions is negative, and surface-active cations are strongly adsorbed from such solutions. If the pH of the solution falls, the surface potential<sup>19</sup> decreases, and, below the isoelectric point, the potential becomes positive. Thus it seems probable that adsorption of surface-active cations by wool from acid solutions below the isoelectric point is due to interaction between non-polar parts of the fibre and non-polar parts of the cation.

Adsorption of non-ionic surfactants of the alkylaryl polyethylene oxide type probably occurs also through non-polar forces. Addition of acid slightly increases adsorption, an effect similar to that obtained on adding salts to such surfactants<sup>20</sup>.

Crewther, in his investigations into the effect of surfactants in carbonising<sup>8</sup>, found that, whereas the amounts of acid adsorbed from a solution containing acid and from one containing both acid and a setting agent may be the same, wool immersed in the second solution suffered less degradation. Non-uniformity of acid distribution, in absence of wetting agent, is likely to cause localised damage, resulting in lower "tear", even when the amounts of acid retained by the wool are similar to those retained from a solution containing a wetting agent. Surfactants such as condensates of ethylene oxide and propylene oxide, whose large molecular size may not permit them to penetrate to the less accessible portions of the fibre, where hydrogen ions can penetrate, will not be so effective in promoting uniform distribution of acid. They are, therefore, not effective in improving tear<sup>21</sup>, just as they appear to have no effect on the rate of adsorption of sulphuric acid. Non-ionic surfactants of lower molecular weight, on the other hand, increase tear<sup>22</sup>, probably because of their more thorough internal, as well as external, wetting action. The concentrations of sulphuric acid used in carbonising baths are, of course, considerably greater than those employed in the present work.

When the quantity of acid in the bath is less than that required to saturate wool, the role of adsorbed hydrogen ions in determining the distribution of anions in the fibre becomes important.

The combination of acid with wool can be considered to take place in two rapidly successive stages. Immediately after the fibre is immersed, hydrogen ions, owing to their great mobility, will combine with the fibre at suitable sites, such as the salt linkage, to be followed by the less mobile sulphate ions, to complete the second stage<sup>23</sup>—



Thus the electrical neutrality of the fibre is restored.

According to modern views on the mechanism of dyeing of wool with levelling acid dyes, dyeing then proceeds by gradual displacement of sulphate ions by dye anions, because of their greater

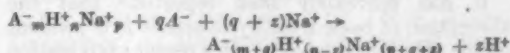
affinity. Thus, the ultimate distribution of sulphate and dye ions depends on the distribution of positively charged sites; therefore, uniform distribution of these sites will assist level dyeing. If greater uniformity can be achieved during the stage immediately after immersion of the fibre in the bath, the time required subsequently to achieve uniformity will be reduced.

Hydrogen ions are very rapidly adsorbed by wool, and it therefore seems desirable to slow down adsorption so as to minimise acid concentration gradients between and within fibres. Addition of anionic surfactants to the acid bath appears to afford such a means of control.

Surface-active anions attract positively charged hydrogen ions and, having a much lower mobility, reduce the mobility of hydrogen ions. One would therefore expect some correlation between the mobility of surfactant anions and the effect on the rate of adsorption of hydrogen ions. If it is assumed that there is a correlation between ionic size and mobility, bulkier anions should have a greater effect in reducing the rate of adsorption of hydrogen ions. Anionic surfactants which have a low critical micelle concentration are far more effective in this respect than agents with high critical micelle concentrations. This is probably because of the greater bulkiness of anionic micelles as compared with single anions, as is shown by the relatively slow rate of adsorption of anionic surfactants at concentrations higher than their critical micelle concentration.

A strange feature in the general pattern became evident when sodium sulphate was added to acid solutions containing anionic surfactants. For example, sodium dodecylbenzenesulphonate, which retards acid adsorption very effectively, loses its effect almost entirely if sodium sulphate is added, and acid adsorption becomes rapid again, whereas the rate of adsorption of surface-active anions is reduced. The reason for this behaviour is not known. It is suggested that the addition of a large quantity of sodium ions causes a reduction in the critical micelle concentration<sup>18</sup> through two mechanisms:

- (i) free surfactant anions aggregate into micelles, and
- (ii) a shift in the equilibrium occurs:



where A<sup>-</sup> represents a surface-active anion and



are anionic micelles with different proportions of hydrogen and sodium ions being bound to the micelle in which  $m > (n+p)$ . Such a shift results in an increase in the size of the micelle and it may cause a simultaneous release of hydrogen ions from the micelle. Additional work is in progress to seek an explanation of this phenomenon.

The authors wish to thank the Australian Wool Research Committee for a grant, their colleague, Dr. C. H. Nicholls, for useful discussions, and the



firms who generously provided the chemicals used in this work.

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(Received 22nd August 1960)

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## CORRESPONDENCE

*The Editor does not hold himself responsible for opinions expressed by correspondents*

### Extraction of Dyes from Acrylic Fibres

In a recent paper<sup>1</sup> Brown has drawn attention to the difficulty of extracting dyes from acrylic fibres. We have found that a 50/50 (v/v) mixture of nitromethane and methyl n-propyl ketone (2-pentanone) is a useful extracting solvent at 90–100°C. Both disperse and basic dyes are extracted from Courtelle, and the former can be removed quantitatively by repeated extraction. To achieve more nearly quantitative extraction of basic dyes, it is necessary to add 10% (v/v) of acetic acid to the extraction mixture.

Certain dyes appear to be unstable in this solvent mixture, but the majority of those which we have used have been unaffected.

D. BLACKBURN  
K. MELDRUM

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DROYLSDEN  
MANCHESTER

20th October 1960

<sup>1</sup> Brown, *J. C.*, *J.S.D.C.*, **76**, 539 (1960).

### A Simple Dyeing Test to Distinguish between Courtelle, Orlon Type 42, and Standard Acrilan

It has previously been reported<sup>1</sup> that the absorption of basic dyes by Courtelle is dependent on the pH of the dyebath. In a recent examination of this method of controlling the rate of absorption of basic dyes by Courtelle, an indicator effect was observed with the dye Sevron Orange L (DuP). Under neutral or weakly acid conditions, the colour of an aqueous solution of this dye is orange, but when sulphuric acid is added the solution

becomes red. If Courtelle is treated in the latter solution for 15 min at the boil, a pale orange colour is obtained, whereas under the same conditions a red colour is produced on Orlon Type 42.

A simple dyeing test to differentiate between Orlon Type 42, Standard Acrilan and Courtelle involves the use of Sevron Orange L in presence of sulphuric acid, together with a level-dyeing acid dye (e.g. Lissamine Green SFS—ICI), and a non-ionic anti-precipitant (e.g. Lubrol W—ICI).

The recommended procedure is as follows—  
A sample of the unknown acrylic fibre is treated for 15 min at the boil in 40 volumes with—

- 2% Sevron Orange L
- 2% Lissamine Green SFS (all percentages based on weight of fibre)
- 4% sulphuric acid
- 1 g/l. Lubrol W

The resultant colour is—

- Orlon Type 42—red
- Standard Acrilan—green
- Courtelle—pale orange

It is advisable to include small samples of these fibres in the dyebath together with the fibre being tested. This precaution is particularly recommended if the unknown acrylic fibre is coloured and requires stripping before the test is applied. In this case the control sample should be included in both stripping and re-dyeing stages.

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20th October 1960

<sup>1</sup> Meldrum, K., and Ward, J. S., *J.S.D.C.*, **74**, 140 (1958).



## Notes

### Society Honours

At its meeting on 2nd November 1960 Council made the following awards—

<i>Gold Medal</i>	W. A. Edwards W. Penn
<i>Silver Medal</i>	R. Grice E. I. Noble C. B. Stevens
<i>Bronze Medal</i>	F. Harrison

Presentation of these awards, the citations for which appear in the appropriate Medals and Awards notices on pp. 5-6 of this issue, will take place at a special ceremony (see below).

### Worshipful Company of Dyers Research Medal

The Research Medal of the Worshipful Company of Dyers of the City of London has been awarded to Dr. J. Wegmann for a paper on "Effect of Structure on the Change in Colour of Vat Dyes on Soaping", published in the *Journal* in May 1960.

### Presentation of Medals and Diplomas

Council, at its meeting on 2nd November 1960, decided to hold a special ceremony for the presentation of the Society's Awards, including Medals and Diplomas. This ceremony will follow the Council meeting on 1st March 1961 in the main restaurant of the Victoria Hotel, Bridge Street, Bradford, at 4 for 4.15 p.m., when afternoon tea will be served. It is expected that representatives of the industrial and academic fields, including those responsible for the training and education of candidates for the Associateship, will be present. Relatives and employers of those receiving awards will also be welcome.

### New Year Honours

In the New Year Honours, Mr. Alan Herries Wilson, M.A., F.R.S., a Deputy Chairman and Managing Director of Courtaulds Ltd, and a member of the Society since 1946, received a knighthood.

### Abbreviations and Symbols

The Society's Publications Committee has decided that from January 1961 the style to be used in the *Journal* for abbreviations, etc., shall be that of B.S. 991: Part 1: 1954 "Letter Symbols, Signs and Abbreviations". This standard is divided into three sections—general principles, specific usages, and indexes. The second section lists all the symbols likely to be encountered under the headings—(a) mathematical operations and constants, (b) physical quantities, (c) chemical elements, (d) names of units, and (e) words other than names of units. Relevant abbreviations and symbols will be listed in the Index to the 1961 *Journal*, which will be published early in 1962. Authors of papers and abstractors are asked to adopt the recommendations of B.S. 991 in their manuscripts.

### A.S.D.C. Examination 1961

The annual examination for the Associateship of the Society of Dyers and Colourists will be held on Thursday, Friday, and Saturday, 1st, 2nd, and 3rd June 1961. Candidates may take Papers A and B together, followed in a subsequent year by C, D, and either E(i) or E(ii); or Papers A, B, and C may be taken together, and D and either E(i) or E(ii) subsequently. Candidates must indicate the special branch of tinctorial technology in which they desire to be examined and state whether they intend to take the examination in one or two stages, and which of the two papers E(i) and E(ii) they will take.

Application forms can be obtained from the General Secretary at the Society's offices, and must be completed and returned not later than 28th February 1961.

### Colour Index Additions and Amendments

Publication of additions and amendments to the *Colour Index* will continue quarterly in 1961. The subscription for the four issues will be £2. Further particulars may be obtained from the General Secretary.

### Half-day Symposia

Two successful half-day symposia have recently been held by Sections of the Society. On 9th December about 100 people attended the West Riding Section's symposium on "New Dyeing Techniques" at the Bradford Institute of Technology. Papers were given on the high-temperature beam dyeing of Terylene fabrics, production of fast dyes on polyester fibres and blends, a continuous dyeing process for wool using formic acid, and the acid shock process. One of the speakers, M. Marian (CFMC), conveyed greetings to the Society from M. Zuber, President of the French Association of Textile Industry Chemists (ACIT).

A similar number attended the symposium on "Chemical Finishing" organised by the Midlands Section and held at Daybrook House, Nottingham on 15th December. The subjects covered were "Stimulus and Response", the chemistry of resins, lubricants in resin finishing, and production of easy-care finishes on viscose rayon.

### Symposium on New Developments in Plastics

A symposium on "New Developments in Plastics" will be held on Saturday, 11th February 1961, at the Bradford Institute of Technology. The symposium is being organised by the Bradford Chemical Society, The Plastics Institute (Yorkshire Section) and the Department of Chemical Technology of the Institute, in conjunction with the Yorkshire Council for Further Education. Members of other Societies are also invited to attend; all enquiries should be addressed to Dr. R. P. Sheldon, Department of Chemical Technology, Bradford Institute of Technology. The subjects to be discussed are "The Nature and Properties of Plastics", "New Plastics", "The Effects of Structural Modifications on the Properties of Polyvinyl Chloride" and "The New Approach of Fast Speed and Fully Automatic Moulding of Thermosets".

### Biochemical Society 50th Anniversary

To celebrate its fiftieth anniversary the Biochemical Society is holding a meeting at University College, London, on 27th-29th March 1961. A symposium on "Structure and Synthesis of Macromolecules" will be held on 27th-28th March at Senate House, University of London. The morning of 29th March will be devoted to original communications at University College. Non-members may attend these functions.

### 1st International Congress on Metallic Corrosion

This congress will be held at the Imperial College of Science and Technology, South Kensington, London S.W.7 on 10th-15th April 1961. A detailed programme appeared in *Chem. and Ind.*, No. 48 (26 Nov 1960) 1473, and further information is available from the Honorary Secretary of the Executive Committee, 14 Belgrave Square, London S.W.1.

### R.I.C. Annual Conference

The 1961 Annual Conference of the Royal Institute of Chemistry will be held in Southampton on 20th-22nd April, and will include a one-day symposium on "Chemicals from Petroleum". Accommodation is limited, so that members who wish to attend should make early application on a form obtainable from the Institute.

### Technology of the Textile Industry (U.S.S.R.)

Publication of an English cover-to-cover translation of the Russian journal *Tekhnologiya tekstil'noi promyshlennosti* under the above title will commence early in 1961. The first issue to be translated will

be that of Jan/Feb 1960, which will be available in Feb/Mar 1961. The translation is sponsored by the D.S.I.R. and the journal will be edited by specialists in organisations associated with the ASLIB Textile Group under the general editorship of V. D. Freedland. The journal will be published every alternate month, and the annual subscription will be £7 (\$21.00) including postage. A reduction of 25% is allowed to libraries of universities and technical colleges. The publishers are The Textile Institute, 10 Blackfriars Street, Manchester 3.

### Patents Act, 1949, and Registered Designs Act, 1949

On 15th November last the Patents and Designs (Renewals, Extensions and Fees) Bill had its second reading in the House of Lords.

The primary purpose of the Bill is to enable the Patent Office to extend from three months to six, the period in which the proprietor of a patent may apply after the expiration of the current period of protection, for a renewal and to provide for a like period of grace to make application for the extension of copyright of a design.

Opportunity is also taken by the Bill to clear up a point of obscurity in the Registered Designs Act, 1949, and also to permit the Patent Office to charge higher fees in relation to patents. It was stated in the House of Lords that the only increases which it was proposed to make were to increase the application fee for a patent from £1 to £2 and the fee for the filing of a complete specification from £4 to £8.

Some adverse criticism was raised in the debate in the House of Lords, on the procedure by which it was proposed to increase the fees. The Bill proposes to repeal the first schedule to the Act which prescribes certain maxima for the fees.

L. E. JONES

### Death of Members

We regret to report the death of Dr. C. J. T. Cronshaw, a Past President of the Society, Mr. Dick Carter, Vice-Chairman of the West of England and South Wales Section, and Mr. H. E. J. Cory.

### Meetings of Council and Committees December

Fastness Tests Co-ordinating— 6th  
Finance and General Purposes— 13th  
Publications— 13th  
Diplomas— 14th  
Colour Index— 16th

## OBITUARY NOTICE

### Alec Bowring Steven

A. B. Steven, who died on 11th October 1960, at the age of 83, was the first holder of the Graham Young Lectureship in Dyeing at the (now) Royal

College of Science and Technology, Glasgow. Appointed in 1908, he held the position for 37 years. A student of Sir William Ramsay at University College, and a graduate of London

University, he went to Glasgow with a distinguished academic record and some lecturing experience at Leeds.

The classes at Glasgow were at first small and largely practical in content. The most significant development during Steven's tenure of office was their gradual extension to full-time day courses. Dyes and Their Application became an optional subject in the final year for the Honours Degree in Applied Chemistry of the University of Glasgow, with which the College became affiliated in 1913. In less than ten years Steven was largely instrumental in raising the subject to full university level. The classes attracted more than half the students in, at the time, the largest school of applied chemistry in the country. This fact did much to ensure a continuance of the Lectureship on his retirement.

From the outset Steven was an extremely popular member of the staff. His gifts of leadership were early recognised. If a committee was set up to consider and report upon any problem affecting the staff, Steven was the obvious chairman. Many of the improvements that they enjoy

today were the result of Steven's leadership in staff matters. In his efforts to improve the social atmosphere of the College he was ably supported by his wife, whose tragic death by accident a few years ago was a severe blow to him.

During the last war Steven was a member of a team under the Explosives Directorate of the Ministry of Supply and made useful contributions to a wide range of problems. For a time between the two wars he acted as Head of the Chemistry Department at St. Mungo's Medical School in Glasgow, undertaking this additional work without any sacrifice of his own department.

The formation of the Scottish Section of the Society owes much to Steven. He was a founder member from 1908 until 1935, and from 1917 to 1920 he held the post of Honorary Secretary.

Although he did not make many contributions to the technical development of his subject, Steven will long be remembered as a worthy pioneer in its teaching, both theoretical and practical.

W. M. CUMMING

## New Books and Publications

### Deutscher Färbekalender 1960

(Volume 64)

Edited by O. Mecheels. Pp. iv + Calendar + 384 + iv half-tones + 60. Munich: Franz Eder Verlag. Price, DM 15.50.

This volume follows the pattern of its predecessors, i.e. it contains a miscellany of papers followed by several reference sections. The former includes articles on the physics of colour, various dyeing processes (particularly relating to man-made fibres and levelling agents), resin finishing, dry-cleaning, and light-fastness testing. Each article will have its own appeal for the specialist, but most will also provide stimuli for the dyer because of their topicality.

The reference sections consist of tables of man-made fibres, dyes, and specialities, and a commercial guide. The current volume maintains the high level of competency that has come to be expected of this work.

S. R. COCKETT

### A Practical Manual of Industrial Finishes on Wood, Metal and other Surfaces

By B. M. Letsky. Pp. xii + 251 + 17 half-tone pp. London: Chapman & Hall Ltd. 1960. Price, 35s. 0d.

As the title of this book makes clear, it deals with a class of finish not normally encountered by the cloth finisher, at least in the way of business. Nevertheless, on the principle that it is often extremely useful and stimulating to learn a little about another man's technology, this book should prove interesting to such readers of the *Journal*. Industrial finishes for wood and metal, i.e. paints, varnishes and lacquers, now embrace a wide range of polymeric types, some of them familiar to cloth

finishers, but many of them specific to the paint industry. In the past, nearly all wood and metal finishes have been of the type soluble in organic solvents, and hence of little interest to cloth finishers, apart from those in the coating industry. Now there is growing interest in the paint industry in water-based finishes, some of them very similar to those long used in the treatment of textiles, and it is to be hoped that increasingly each industry may learn from the other.

Inevitably in a book of this length, dealing with a subject of such breadth and complexity, the treatment will not satisfy the specialist. But as a readable and fairly comprehensive account of the present status for non-specialists, this book can be warmly recommended. Its usefulness would, however, have been considerably increased by the provision of a bibliography.

L. VALENTINE

### An Introduction to the Organic Chemistry of High Polymers

By Carl S. Marvel. Pp. viii + 82. New York and London: John Wiley & Sons Inc. and Chapman & Hall Ltd. 1959. Price, 36s. 0d.

Many excellent books already exist that are suitable for the research worker in high polymer chemistry and the plastics technologist, but relatively few have been written for the undergraduate who desires to co-ordinate the organic chemistry of high polymers with his more general studies. For that matter, there is little written for the older chemist experienced in fields of a totally different nature, who wishes to find out quickly what polymer chemistry is about. This particular book has been written with the clear intention of meeting this need, and as such it



should be found to be a stimulating account of one of the major developments of organic chemistry.

All the well-known polymers and methods of polymerisation are briefly covered, but entirely from the organic chemist's point of view, the physical chemistry receiving little attention. Of necessity in such a short book, the space allotted to individual polymers is relatively small, and the extent of treatment has been determined more by the chemistry involved than by commercial importance.

In general the treatment is up-to-date, but there are fields where this is not so. An example of this concerns the condensation of urea and formaldehyde, where the work of the last ten years associated with, among others, de Jong and de Jonge, Zigeuner, and Fahrenhorst is not mentioned. It could be argued, however, that matters which are still controversial are best omitted from an introductory volume.

The book is fairly well referenced, with a tendency to give most references to the most recent developments, such as ionic catalysts, block polymers, etc. Mistakes are few, although the omission of an arrow makes an equation on page 20 confusing at first sight.

Although this book could be warmly recommended to the student, such a small monograph at such a high price is not likely to commend itself to his pocket.

W. S. SHORT

#### **Handleiding voor het Toepassen van Spectrofotometrie en Kleurmeting**

(Manual for the Application of Spectrophotometry in Colour Measurement)

Mededeling No. 116 van het Vezelinstituut T.N.O.  
By L. F. C. Friele. Pp. vi + 53 + VII Tables + 14 Figures. Delft, Holland: Vezelinstituut T.N.O. 1958.

This publication originates from the Netherlands Fibre Research Institute (Vezelinstituut T.N.O.) in Delft. This Institute, amongst its manifold activities concerned with fibres and textile materials, has been making advanced studies in the field of colour measurement for many years. Indeed, as long ago as the immediate post-war years, precise work on reflection spectrophotometry of fabrics was in progress. It is interesting to see that some of the relevant data used in the Vezelinstituut for their work on dyed textile materials have been collected together in this monograph.

The monograph sets out to be "a guide for application of spectrophotometry and colour measurement". It includes a discussion of transmission and reflection spectrophotometry, and there are several lengthy tables, each occupying several pages, of, e.g., the Kubelka-Munk and Nochel-Stearns functions for reflected light from dyed textiles. There is also an extensive table giving costs and performance data of a number of spectrophotometers, which could be useful to anyone intending to purchase an instrument.

An account of the system of colour measurement used by the International Commission on Illumination (C.I.E.) is given and, in addition, there are tables and graphs dealing with the effect of, e.g., concentration and mixtures of colouring matters in dyed textiles on the colour of the resulting material.

An English summary of the text is included, but as it occupies only 1% of the text it does no more than whet one's appetite for a meal which most non-Dutch readers cannot digest, for the text, as a whole, is in Dutch. Even with that grave handicap for English readers, specialists in the field of spectrophotometry of fabrics may find that the tables and graphs will be of use, since it is not too laborious a task to translate the legends.

J. M. PRESTON

#### **Chromatographic Reviews 1959**

(Volume 2)

Edited by M. Lederer. Pp. viii + 195. Amsterdam: Elsevier Publishing Co. 1960. Price, 46s. 0d.

This book follows the style of Volume I of the series in that it consists of reviews that have previously been published in the *Journal of Chromatography*. Those not originally in English have now been translated. There are six reviews in this volume; they deal with "Gas-Liquid Chromatography", "Starch Block Electrophoresis", "Paper Chromatography of Dinitrophenylamino Acids", "Chromatography of Flavonoid Pigments", "Separation of Different Types of Human Haemoglobin", and "Inorganic Adsorption and Precipitation Chromatography". A very useful subject index is included.

In the conclusion to his article on "Chromatography of Flavonoid Pigments", J. B. Harborne discusses briefly the advantages and disadvantages of chromatography as a means of identification. On the one hand, it provides a rapid method using only small amounts (a few mg) of material, it yields a new physical constant (the  $R_f$  or  $R_m$  value) comparable with melting point as a means of identification, and it will separate complex mixtures, such as the pigment mixtures found in cultivated plants. On the other, it must always be used in conjunction with other appropriate analytical methods, there is a definite limit to its resolving power, and its value is often limited in the identification of substances (e.g. flavonoids) of complex structure (e.g. bisflavonoids).

This volume should prove of great interest to workers in chromatography and related fields.

M. T.

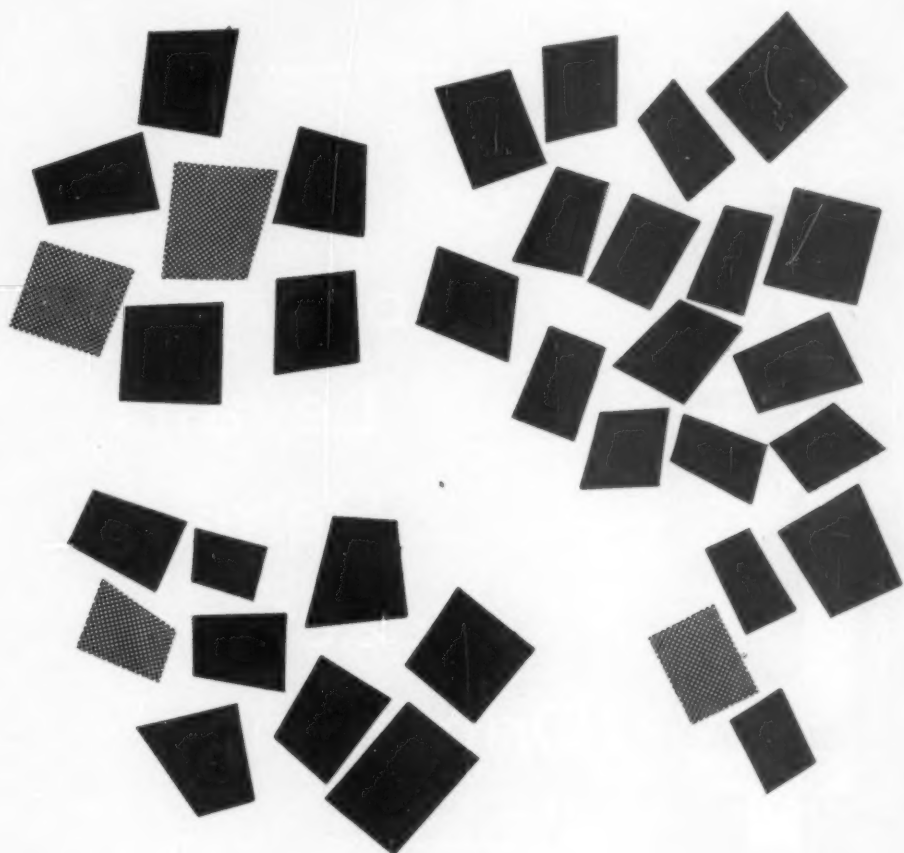
#### **Mechanics for Textile Students**

By W. A. Hanton. Pp. x + 336. Manchester and London: The Textile Institute and Butterworths. 1960. Price, 30s. 0d.

This is a photolithographic reprint of the book originally published by the Textile Institute in 1954. The book is divided into three parts, dealing respectively with the study of movement, statics, and applications of force in causing motion. Many examples are included, the majority of them being concerned with textile machines and processes.

M. T.





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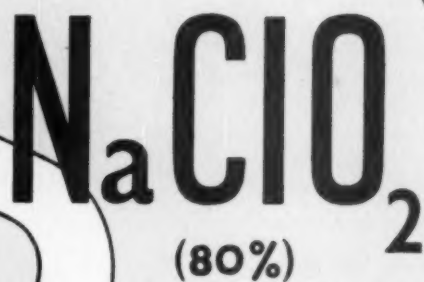
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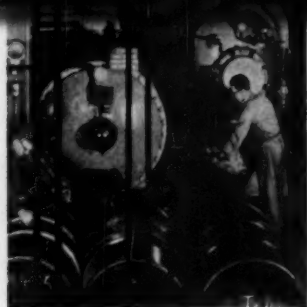
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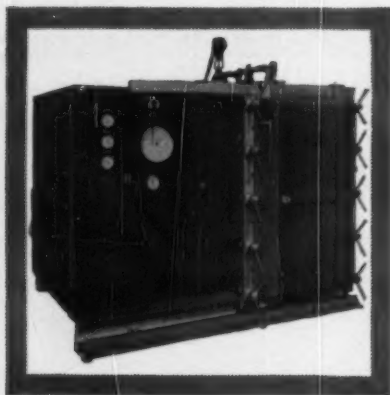
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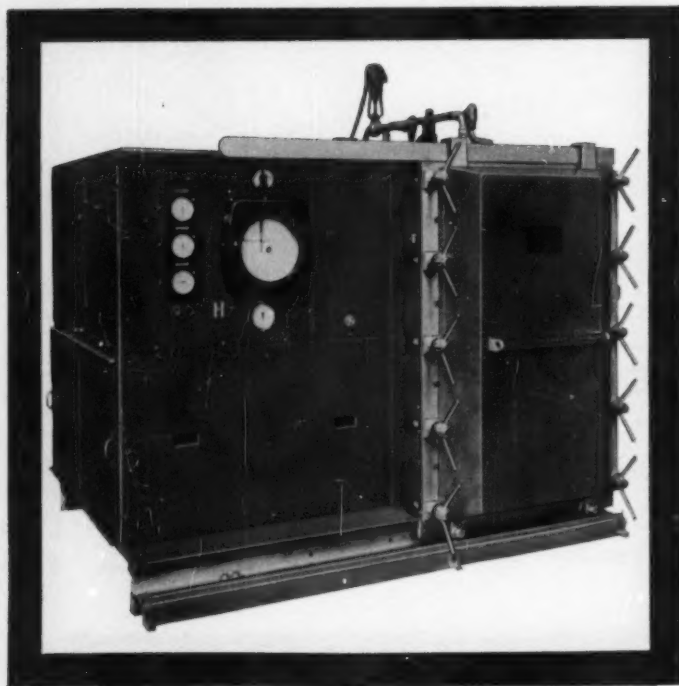
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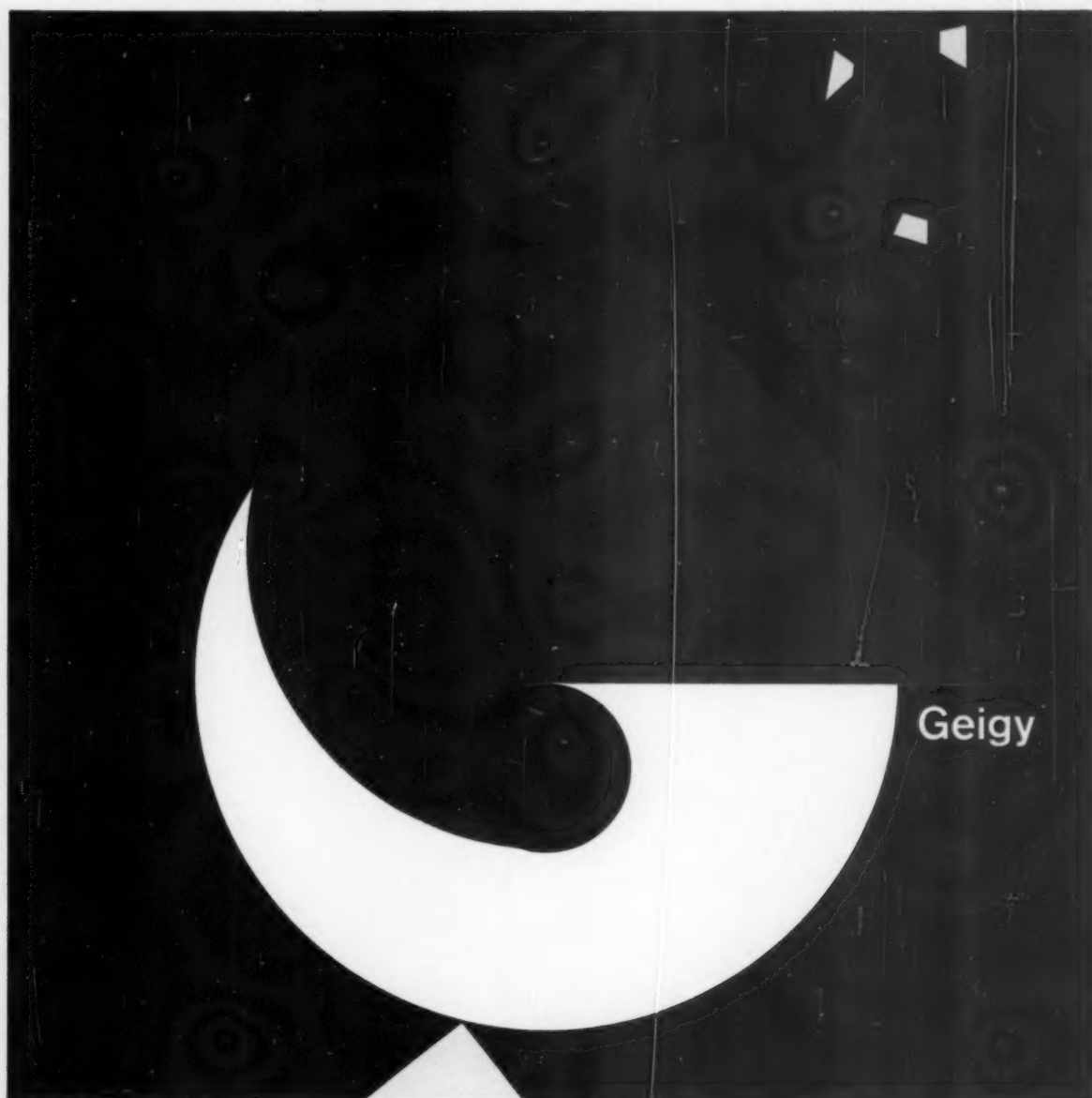
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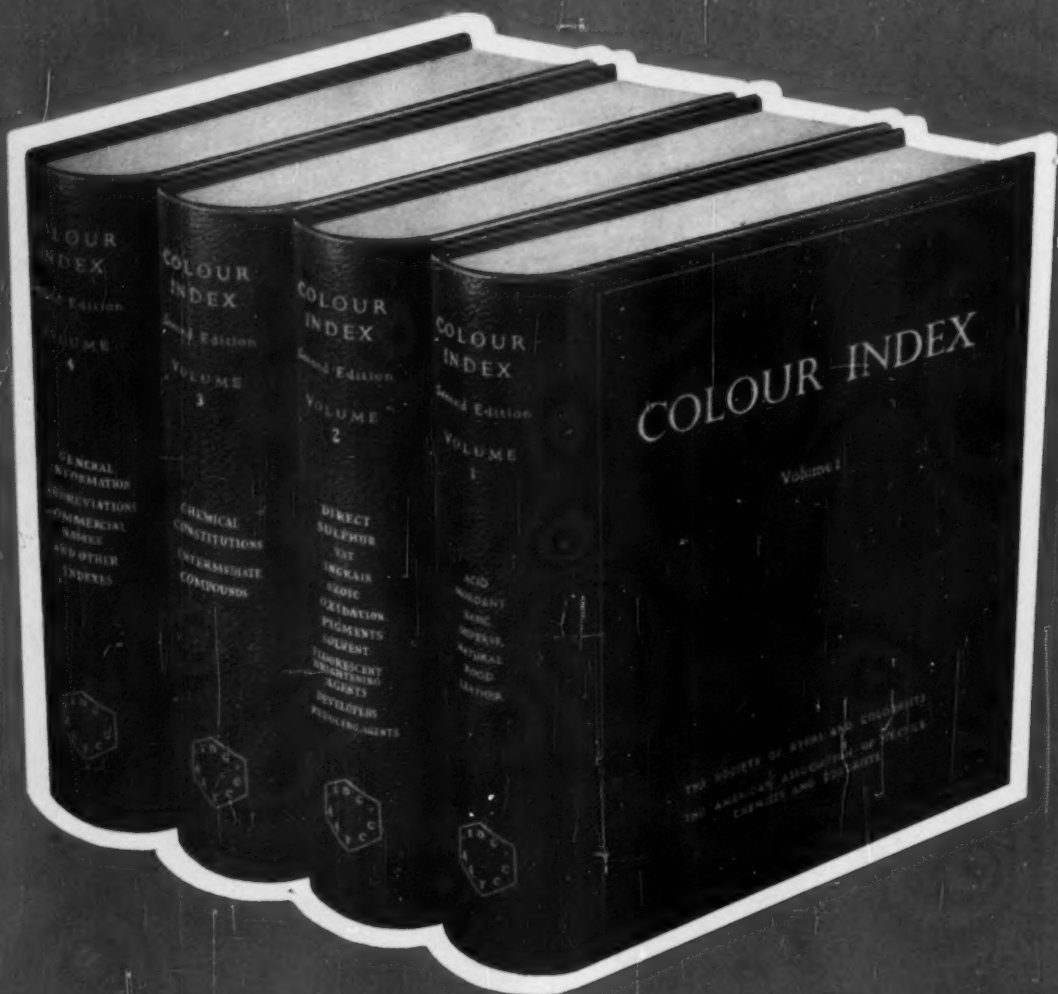
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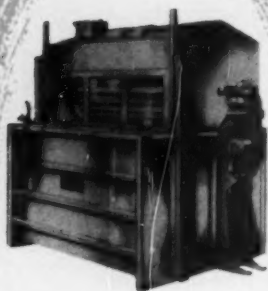
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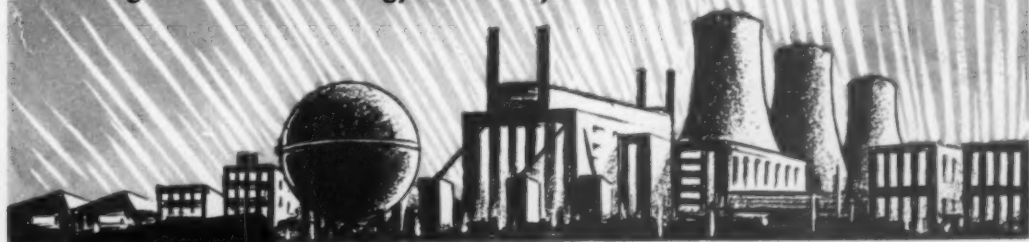
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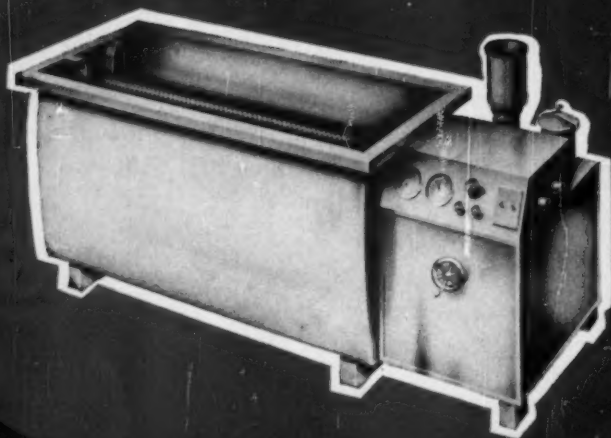
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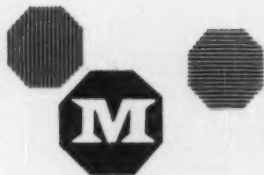
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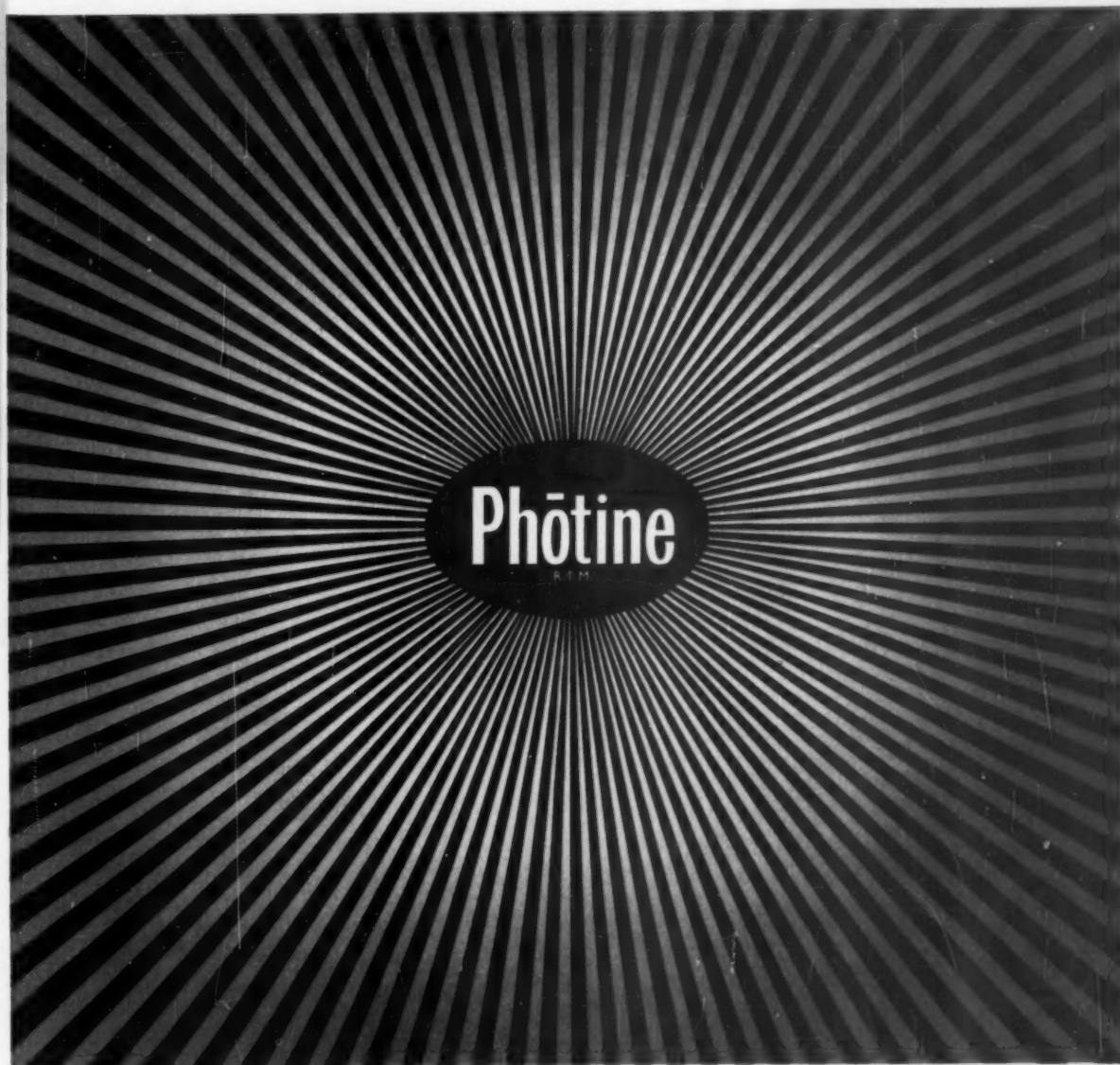
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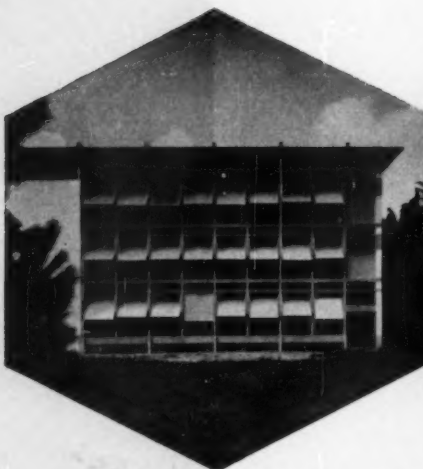
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# Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index

## I—PLANT; MACHINERY; BUILDINGS

### Performance of Titanium Dioxide Pigments in a Sand Grinder

G. Brownlie

J. Oil & Col. Chem. Assoc., 43 (Oct 1960) 737-761

The satisfactory performance of a sand grinder depends upon the selection of a suitable grinding charge; Ottawa sand and glass ballotini of suitable size are satisfactory. Optimum conditions for continuous processing require a mill-base of low viscosity and of flow characteristics that are approximately Newtonian. Milling time should be minimised to avoid possible discoloration of the product. Modern titanias lend themselves readily to sand grinding. There are several photographs. J.W.D.

### Water Mangle

Fibres & Plastics, 21 (Nov 1960) 346-347

A Perlon bowl, sealed on all sides, floats on a cushion of water formed within an Al pressure chamber. During running, water pressure is applied to the projected area of this bowl, forcing it against an ebonite counter-bowl. Even pressure distribution is obtained between the bowl faces, and no camber is required. The standard machine caters for working widths from 36 in. to 126 in. and has a pressure capacity of 57 lb/in<sup>2</sup>. J.W.D.

## PATENTS

### Carbon Black (C.I. Pigment Black 7) Furnace

Phillips Petroleum Co. BP 849,169 (U.S.A. 6 Apr 1956)

### Powder of Uniform Particle Size

ICI BP 851,067 (15 Jan 1958)

A powder composed of particles of wide size distribution may be resolved into fractions of limited size distribution by suspending it in a suitable liquid which is passed up a column by a re-circulating system; the required fraction may be removed continuously from a selected level in the column. Uniform flow is ensured by packing the bottom of the column with dense particles, e.g. small glass beads (ballotini). E.S.

### Mill for Ink, Paint, and the like

J. H. J. Wood BP 848,514 (31 July 1958)

The vehicle and pigment are ground together in the annular space formed between an outer cylinder and an inner rotor. This space contains salts or other grinding assistants and has deflecting elements arranged lengthwise of the rotor and mounted radially on it. The surfaces of these elements come close to the outer cylinders so that the grinding assistants and the material being treated are agitated and dispersed as it passes from one end to the other of the annular space. C.O.C.

### Wet Processing of Loose Textile Material

B. Thies K. G. Spezialmaschinenfabrik

BP 848,963 (Germany 15 May 1957)

Two cylindrical containers are placed with their axes vertical coaxially one above the other around a central delivery passage. Each container has an impermeable cylindrical outer side wall and permeable top and bottom walls. There is a space between the bottom of the higher and the top of the lower container. This space forms an annular liquid-distribution chamber having an impermeable side wall and receives liquid from the central delivery passage. This ensures even treatment of the material in the containers. C.O.C.

### Feeding Loose Fibres to Drum Dryers

Fleissner & Sohn Maschinenfabrik

BP 849,835 (Germany 8 June 1956)

### Winding a Web Backwards and Forwards from One Roller to Another

C. A. Brown

BP 850,387 (16 Feb 1957)

### Controlling Tension in a Running Web

Cameron Machine Co.

BP 843,898 (10 Nov 1958)

A brake on the pay-out roller is operated by compressed air and is continuously subjected to very fine control in response to variations in force applied by the web to a guide roller over which it passes. C.O.C.

### Continuous Open-width Treatment of Fabric

Mezzara

BP 848,537 (Italy 26 Feb 1958)

In a machine of the type described in BP 793,283 (J.S.D.C., 74 (1958) 557) the cross-sectional area of the outlet opening in the receptacle supplying the fabric to the vat can be varied simultaneously throughout its length to suit fabrics of various thicknesses. The means for doing this are independent of the means for varying the cross-sectional area to restrict it in proximity to its longitudinal central plane. C.O.C.

### Transportable Boxes for Holding Webs wound on Rollers

E. Kusters

BP 849,764 (Brazil 31 July 1958)

### Controlling Moisture Content of Material passing through a Conditioning Chamber

H. Eicker

BP 847,537 (Switzerland 26 Oct 1956)

### Dry-cleaning Apparatus

Böhler & Weber

BP 847,809 (Germany 2 Dec 1955)

Plant containing two drums and so controlled that while drying is being done in one drum, washing or centrifuging is being done in the other.

BP 847,808 (Germany 15 July 1955)

Automatic controls for the above plant are described. C.O.C.

### Pads for Platens of Steam-heated Garment Presses

Isaac Braithwaite & Son (Drysalts) and Dunlop Rubber Co.

BP 848,662 (29 July 1955)

A resilient pad for one of the platens of a garment press is formed from silicone foam rubber and pressed in absence of heat till it attains constant thickness. Such pads have a long working life. C.O.C.

### Hosiery Finishing Machine

E. E. Bellmann, R. Kronsbein, and F. Bellmann

BP 847,274 (Germany 18 July 1956)

A machine for preboarding, dyeing and finishing synthetic-fibre hosiery comprises two adjacent circular conveyor tracks carrying the metal hose forms. The tracks are led through a common pressure vessel for simultaneous admission of a batch of forms from each track, the vessel then being sealed for application of steam pressure and of dye or finish solution by spraying. The conveyor tracks also pass through a zone in which further spray application may be carried out and the hosiery dried. Sufficient forms are carried by the conveyors to enable unloading and reloading to take place whilst batches are being treated. G.E.K.

### Multi-colour Printing Machine

Mather & Platt

BP 851,168 (3 Mar 1958)

Improved and simplified means for obtaining correct register of the several printing rollers. C.O.C.

### Printing Machine

Morrison Machine Co. BP 844,557 (U.S.A. 16 June 1958)

A printing machine having more than one cylindrical screen in which the printing paste supplied to and maintained within each cylinder and the pressure exerted on material passing between each cylindrical screen and a pressure roller may be accurately controlled and adjusted while the machine is working. C.O.C.

### Multi-colour Printing on Stretchable Fabrics

Plastic Film Corp.

USP 2,921,517 (7 July 1955)

A machine for printing resin-based inks on stretchable knitted or woven fabric, e.g. sheer nylons or wool fabrics, with accurate registration of the different colours. C.O.C.

### Printing Tables for Air-impermeable Foils

Fritz Buser

BP 851,167 (26 Feb 1958)

Below the surface of the table tops and of the feed carriage there are passages extending transversely to the direction of movement of the carriage. The passages are parallel to one another and each opens out on the surface of the table by a series of suction apertures running in the direction of the passage. Each passage has a pneumatic feed pipe controlled by a valve. This enables the foil to be kept firmly in contact with the table. C.O.C.

**Hydraulic Loading of Printing Rollers**

Mather &amp; Platt BP 852,429 (19 Dec 1957)

Improved means for obtaining uniformity of movement of the ends of the printing roller as it approaches and engages the impression cylinder, the load being increased after such engagement. The hydraulic loading means then returns the printing roller to its original position out of contact with the cylinder.

C.O.C.

**Combined Coating and Printing Machine**

E. A. Timson BP 850,948 (21 Jan 1958)

A machine suitable for printing on flimsy, flexible material first coats the material, dries it and then prints on it.

C.O.C.

**Warp Sizing Machine**

Ruti Machinery Works

BP 850,485 (Switzerland 24 Nov 1956)

Compressed air nozzles blow size from the ends of the squeeze rollers. This prevents oversizing of the selvage threads.

C.O.C.

**Vacuum Slot Hydroextractor**

H. Haas BP 843,806 (Germany 25 Mar 1958)

Drying Apparatus for Regenerated Cellulose or Cellulose Ether Film.

American Viscose Corp.

BP 849,678 (U.S.A. 17 Dec 1956)

Apparatus which enables the vapour-saturated atmosphere close to the hot drying cylinders and the film to be continuously and rapidly removed and replaced by uncontaminated hot air. This enables the temperature of the drying cylinders to be lowered and so decreases the loss in plasticiser vaporised from the film.

C.O.C.

**Doctor for Paper Machine and Similar**

H. E. B. Scott BP 847,173 (9 June 1958)

The doctor blade is retained against forward withdrawal from the holder by spring tongues on the rear edge of the blade or on a member retained in the holder, engaging in a groove in the holder or in the blade.

R.A.

**Calender for High-gloss Finishes**

W. Kumpf BP 845,087 (Germany 9 Feb 1959)

The overflow tank of the oil-filled, electrically heated roller is provided with a water pocket into which condensation water can flow. The oil-supply pipe terminates a little above the bottom of the overflow tank to prevent water flowing back into the roller which is provided with a spiral or screw-like distribution body for the oil filling. The machine is used for paper, synthetic polymer sheeting, and particularly for the glossing of lacquered sheeting.

R.A.

Using Steam-set Printing Inks in a Letterpress Machine (IX p. 44)

Controlling the Application of Size to Yarn (X p. 46)

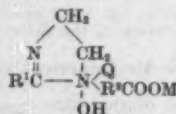
Decorations on Cloth or other Sheet Material (X p. 47)

**III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS****PATENTS****Detergents, Emulsifying Agents, Softeners and Dyeing Assistants**

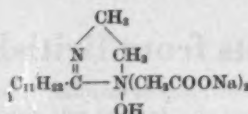
H. S. Mannheimer and J. J. McCabe

BP 853,441 (3 Dec 1956)

Compounds of formula—



(R<sup>1</sup> = hydrocarbon of 4-18 C; Q = R<sup>2</sup>COOM or R<sup>2</sup>O-R<sup>2</sup>COOM; R<sup>2</sup> = aliphatic hydrocarbon of 1-4 C, or hydroxy-substituted aliphatic hydrocarbon of 2-4 C, aliphatic hydrocarbon of 2-4 C containing an ether linkage or a hydroxy-substituted aliphatic hydrocarbon of 3-4 C containing an ether linkage; M = alkali metal) e.g.



are powerful surfactants.

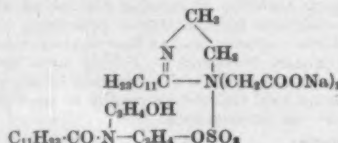
C.O.C.

**Amphoteric Surface-Active Agents**

H. S. Mannheimer and J. J. McCabe

BP 853,440 (3 Dec 1956)

Describes the production of amphoteric surfactants which are anionic at pH > 7. Many general formulae and examples are given, e.g.



They have excellent detergent and emulsifying properties and are excellent dyeing assistants and softening agents for textiles.

C.O.C.

**Solutions of Polyesters**

Chemstrand

BP 853,442 (U.S.A. 9 Jan 1956)

Concentrated solutions of polyesters are obtained by mixing them with an aldehyde (aromatic, cycloaliphatic or heterocyclic) and, if desired, a phenolic compound, and heating to 130°C to b.p. Thus, powdered polyethylene terephthalate (1 g) was mixed with o-methoxybenzaldehyde (9 g) and heated with stirring to 130°C to yield a clear non-gelling solution.

C.O.C.

**Vinylidene Chloride-Acrylonitrile Copolymer Solutions**

American Viscose Corp.

BP 852,866 (U.S.A. 22 July 1958)

A mixture of tetrahydrofuran (50-80% by wt.) and toluene (50-20) is a good solvent for copolymers of vinylidene chloride (< 85% by wt.) and acrylonitrile.

C.O.C.

**Antibacterial Detergent Compositions**

Colgate-Palmolive Co. BP 848,306 (U.S.A. 6 Feb 1958)

A mixture of an anionic or non-ionic detergent (5-50% by wt.), a water-soluble polyphosphate (10-80) and a small amount of 3,5-dichlorosalicyl-3',4'-dichloroanilide is a heavy-duty washing composition having highly effective germicidal activity.

C.O.C.

**Bleach for Use in Detergents**

Unilever

BP 847,702 (7 Mar 1958)

The adducts or "inclusion compounds" obtained by combining urea with organic percarboxylic acids derived from mono- or di-carboxylic acids, and their derivatives, e.g. acyl peroxides, are excellent stable bleaching agents compatible with detergents.

C.O.C.

**Dichlorocyanuric Acid as Bleaching and Disinfecting Agent**

Food Machinery &amp; Chemical Corp.

BP 849,907 (U.S.A. 9 May 1955)

Dichlorocyanuric acid, although very stable during manufacture and storage, is a highly efficient source of active chlorine.

C.O.C.

**Oxidising Wetting and Detergent Compositions**

Colgate-Palmolive Co. BP 848,397 (Canada 14 Apr 1954)

Powdered polychlorocyanuric acid is treated with an olefin having a C:C bond, one of the C atoms being tertiary, in presence of an inert medium containing a water-soluble ionic organic surfactant. The amount of the surfactant used is such as to make 5% by wt. of the final composition. The product is stable during storage but when dissolved in water releases its available chlorine.

C.O.C.

**Bleaching and Oxidising Agents**

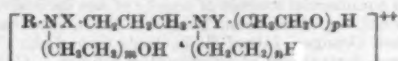
Thomas Hedley &amp; Co. BP 847,566 (U.S.A. 21 Nov 1957)

NN'-Dichlorobenzoyltureas, e.g. 1,3-dichlorotetrahydroquinazoline-2,4-dione, contain a high proportion of available chlorine and are very stable and compatible with most detergents.

C.O.C.

### Polyethoxy Quaternary Ammonium Compounds as Levelling and Stripping Agents

S BP 852,548 (Switzerland 5 Feb 1957)  
Compounds which upon complete dissociation in aqueous solution have a cation of formula—



(R = aliphatic hydrocarbon of 8–22 C; m, n and p each = 0–200 and together = 10–200; X and Y = same or different H, Alk or phenylalkyl) and whose anion is the residue of a non-capillary-active acid, are surfactants having outstanding levelling and stripping capacities for many classes of dye, e.g. direct, acid, or metallised acid dyes. They are also good dispersing agents when pigments are used in dyeing. C.O.C.

### Size for Glass Fibres

Owens-Corning Fiberglass Corp.

BP 848,271 (U.S.A. 9 Apr 1957)

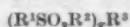
A composition which acts both as a size and as an anchoring agent for resins, especially polyester and epoxy resins, consists of an aqueous system containing polyvinyl acetate (4–12% by wt.), an unsatd. organosilicon compound (0.5–2.0) and a hydrophilic colloid (0.5–2.0). C.O.C.

### Antistatic Agents

Pennsalt Chemicals Corp.

BP 847,106 (U.S.A. 4 Apr 1956)

Sulphones of formula—



(R<sup>1</sup> = Alk of 1–6 C; R<sup>2</sup> = alkylene of 1–6 C; x = 1 or 2; R<sup>3</sup> = OH or acyloxy derived from a satd. aliphatic monocarboxylic acid of 1–17 C when x = 1, or, when x = 2, diacyloxy derived from a satd. aliphatic dicarboxylic acid of 2–10 C or a benzene dicarboxylic acid) are excellent antistatic agents for application to non-cellulosic hydrophobic plastic materials. Thus polyethylene terephthalate fibres treated with 2% of 2-hydroxyethyl-(ethyl)sulphone have excellent antistatic properties. C.O.C.

### Water-soluble Complexes of 8-Hydroxyquinolates

Fran

BP 849,274 (France 25 Nov 1955)

Cu 8-hydroxyquinolate is dissolved in an alcoholic solution of a water-soluble Werner-type complex having a monovalent anion and a cation containing trivalent chromium coordinated with an organic component. The resulting solution is diluted with water.

BP 849,275 (France 25 Nov 1955)

8-Hydroxyquinoline is treated, in one or more stages, with a basic salt of trivalent Cr and a monobasic acid and with a salt of a hydroxide of another polyvalent metal. The product is soluble in water. C.O.C.

### Coating Compositions comprising a Thermoplastic Acrylic Copolymer and a Diglycidyl Ether

Rohm & Haas Co.

BP 848,350 (U.S.A. 20 Aug 1956)

An aqueous dispersion containing an aliphatic diglycidyl ether (which may be water-soluble) and a water-insoluble copolymer of acrylic, methacrylic, itaconic, maleic or fumaric acid with styrene, vinyl acetate or a neutral alkyl ester of one of the acids, on being baked or stoved, yields a hard insoluble film of good gloss and colour and good resistance to heat and chemicals. It is suitable for application, clear or pigmented, to paper, textiles, leather, rubber, wood, metals, etc. C.O.C.

### Dextran-modified Polyester Resins as Modifiers for Urea-Formaldehyde-Alcohol Condensates

Commonwealth Engineering Co. BP 850,534 (3 Oct 1957)

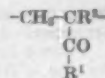
Treating an alcohol-modified urea-formaldehyde condensate with a dextran-modified partly condensed alkyl resin yields a product which hardens on cooling or on being heated to comparatively low temperatures. The product is useful as a coating or impregnating agent and may be applied by dipping, spraying, roller-coating, etc. C.O.C.

### Polymers having Mordant Properties

Kodak

BP 850,281 (U.S.A. 19 Dec 1955)

The product obtained by treating aminoguanidine or a salt thereof (1 part by wt.) with a polymer (0.25–5.0) containing repeating units of formula—



(R<sup>1</sup> = H or Alk of 1–4 C; R<sup>2</sup> = H or, when R<sup>1</sup> = 1, CH<sub>3</sub>) is very strongly basic and soluble in dilute acids. It readily forms water-insoluble salts with acid dyes and is compatible with hydrophilic colloids. C.O.C.

Precipitating Silica (C.I. Pigment White 27) (IV p. 38)

Colophony Adducts as Paper-sizing Agents (XI p. 48)

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

### Halochromism of Alcohols and Dyes

B. N. Dashkevich et al.

*Nauch. Zapiski, Uzhgorod.*

*Gosudarst Univ.*, 22 (1957) 81–108

*Chem. Abs.*, 57 (25 July 1960) 14100

The halochromism of several non-benzenoid unsaturated tertiary alcohols, e.g. subst. 2,5-heptadien-4-ols and their dehydration products (2,5-heptadienes), was investigated. Two new heterocyclic ketones, dye intermediates, were also prepared, viz., 2-acetonilidene-4-hydroxy-1-phenyl-1,2-dihydroquinoline and bis-(4-hydroxy-1-phenyl-1,2-dihydroquinolin-2-ylidene)acetone. A.T.P.

### Stability of Complexes of several Carboxylic Acids formed with Bivalent Metals

M. Yasuda, K. Yamasaki, and H. Ohtaki

*Bull. Chem. Soc. Japan*, 33 (Aug 1960) 1067–1070

Stability constants of the bivalent metal (Pb, Cu, Cd, Zn, and Ni) complexes formed with the mono- and di-carboxylic acids (acetic, benzoic, o-methoxybenzoic, succinic, itaconic, glutaric, adipic, phthalic, malonic, maleic, citraconic, diglycolic, and thiodiglycolic) are determined at 25°C and ionic strength 0.1 by the pH titration method. For monocarboxylic acids and several dicarboxylic acids having no double bond or forming larger chelate rings than six-membered, the following stability order held: Pb > Cu > Cd > Zn > Ni. It is noteworthy that among bivalent metals Pb showed the highest stability. H.H.H.

### Coloration and Decarboxylation of the Aniline Salts of 2,4,6-Trinitrobenzoic Acid in Ketones and other Solvents

M. Hirota

*Bull. Chem. Soc. Japan*, 33 (Aug 1960) 1046–1051

Whereas several aromatic hydrocarbons and phenols form normal molecular complexes with 2,4,6-trinitrobenzoic acid (TNBA), anilines form salts (equimol.) and these salts decompose into the complexes of 1,3,5-trinitrobenzene with the anilines and CO<sub>2</sub> when heated in soln. or in the cryst. state; decomposition is extremely fast in ketonic solvents (in the order acetone > methyl ethyl ketone > ethanol > methanol > dioxan), even at low temp. and with production of striking red colours. An investigation of the relation between coloration and rate of decarboxylation in various solvents has revealed two types of coloration, viz., one which occurs immediately after dissolution in a pure ketone accompanied by rapid decarboxylation and rapid decrease of colour intensity, and another which occurs some time after dissolution in an alcohol, and which gradually increases in intensity when decarboxylation is complete. The spectra of the two types differ in the location of their absorption max. Data are given for aniline, N-methyl- and NN-dimethylaniline, p-toluidine, p-anisidine, p-bromoaniline and pyridine. Since free TNBA recrystallizes from boiling acetone without decomp., it follows that the aniline salt form is essential for the unusual facility of decarboxylation of TNBA in ketones. Nitrobenzene and nitromethane also produce colorations. The order of decarboxylation rates of some p-substituted aniline salts of TNBA is almost parallel to that of the coloration rates, viz., ammonium < p-nitroaniline < p-bromoaniline < aniline < p-anisidine for the coloration, and p-bromoaniline < NN-dimethylaniline < nitroaniline < N-methylaniline < p-toluidine < p-anisidine for the decarboxylation. H.H.H.



### Correlation between Structure and Colour of Organic Compounds. VII—Structure of Azo Dyes

A. A. Kharkharov *Vestnik Leningrad. Univ.*, 12 (4), *Ser. Fiz. i Khim.* (1) (1957) 145-150  
*Chem. Abs.*, 54 (25 Aug 1960) 16833

A new method is suggested for distinguishing between azoid [A(H)]N:NB (I) and hydrazinoid A':NHHB (II) structures of azo dyes. Introduction of electrophilic substituents on B shifts the ultraviolet maximum toward longer wavelengths for I but toward shorter wavelengths for II. With dyes where B = C<sub>6</sub>H<sub>5</sub> (III), 4-nitrophenyl (IV), 2,4-dinitrophenyl (V), 2-nitrophenyl (VI) and 3-carboxy-4-nitrophenyl (VII) and A(H) = radical from 9-isopropylacridine hydrochloride, the following are observed (B, hue, ultraviolet maximum in mμ): (III, yellow, 410), (IV, orange, 460); A(H) = 4-hydroxyphenyl, (III, orange, —), (IV, reddish brown, —), (VII, dark red, —); A(H) = 4-diethylaminophenyl, (III, yellow, 415), (IV, orange, 475), (V, crimson, 520); A(H) = 4-(phenylamino)phenyl, (III, yellow, 412), (IV, orange, 480); A(H) = 2,4,6-trinitrobenzyl, (III, brownish yellow, 375), (VI, reddish brown, 460). For II where A':O is the parent carbonyl compound, then when A':O = 4-nitrobenzaldehyde, (III, bluish red, —), (IV, yellowish red, —) (V, orange, —); A':O = 3-nitrobenzaldehyde, (III, red, —), (V, yellow, —); A':O = 2-nitrobenzaldehyde, (III, deep red, —), (IV, orange, 415), (V, yellow, 375); A':O = 3-nitroacetophenone, (III, orange, —), (IV, yellow, —); A':O = 4-Cl-(CH<sub>2</sub>)<sub>2</sub>N<sup>+</sup>:C<sub>6</sub>H<sub>4</sub>:O, (III, crimson, 519), (IV, bluish red, 508), (VI, red, 500); A':O = 4-Cl-(C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>N<sup>+</sup>:C<sub>6</sub>H<sub>4</sub>:O, (III, crimson, 520), (IV, red, 510), (V, orange, 500); A':O = 4-Cl-C<sub>6</sub>H<sub>4</sub>N<sup>+</sup>:H<sub>2</sub>C<sub>6</sub>H<sub>4</sub>:O, (III, bluish red, 540), (IV, red, 530); A':O = 9-acridinecarboxaldehyde (VIII) hydrochloride, (III, violet red, 562), (IV, cherry red, 520), (V, orange, 500); and A':O = VIII, (III, yellowish brown, 460), (IV, yellow, 444) and (V, yellow, 438). When A':O = VIII, then for A':NN-Na<sup>+</sup>B are observed: (III, greenish yellow, 640), (IV, greenish blue, 630) and (V, violet, —). C.O.C.

### Dyeing Properties of Acid Azo Dyes—V

T. Iijima and M. Sekido

*J. Soc. Text. Cellulose Ind. Japan*, 16 (May 1960) 406-411

Dyes having benzene-azo-benzene and naphthalene-azo-naphthalene structures were converted into the free acids by passing their purified Na salts through a column of ion-exchange resin. Potentiometric titration of the free acids with NaOH gave curves indistinguishable from that obtained with HCl. If the dissociation constant of the sulphonic acid group is K<sub>1</sub>, and that of the OH group K<sub>2</sub>, then K<sub>1</sub> is generally of the order of 10<sup>-2</sup>. In general, for o-hydroxyazo dyes pK<sub>2</sub> ≈ 11 and for p-hydroxy dyes, ca. 8. Amongst the o-hydroxyazo dyes, dyes having 1,4-naphthol-sulphonic acid as coupling component have an anomalous titration curve, e.g. for naphthionic acid-1,4-naphthol-sulphonic acid (NW) acid pK<sub>2</sub> = 8.1, i.e. it accords with the pK<sub>2</sub> of NW acid. This means that the OH group scarcely chelates, although it is in the position *ortho* to the azo group. When dyeing protein or polyamide fibres all the p-hydroxyazo dyes and some o-hydroxyazo dyes in which the OH is not restricted by intra-molecular hydrogen bonding are adsorbed by a mechanism, probably hydrogen bonding with the fibres in addition to the electrostatic attraction between the sulphonic acid group of the dye and the amino group of the fibre. This explains the anomalous dyeing behaviour of these dyes on nylon 6. The standard affinity of some acid azo dyes for nylon 6.6 decreases with increasing acidity of the free dye acid. C.O.C.

### Aromatic Diazo and Azo Compounds. XXXV—Rate of Hydrolysis of Aminoazo Dyes and its Dependence on pH, Substitution and Addition of Ethanol

V. Chmátal and Z. J. Allan

*Collection Czech. Chem. Commun.*, 25 (1960) 210-220

*Chem. Abs.*, 54 (25 Aug 1960) 16140

The rate of hydrolysis of the amino azo dye to the hydroxy azo dye was measured in the whole accessible pH range. The effect of substituents was studied in slightly alkaline and slightly acid solutions and the relation between reaction rate and the Hammett constants σ (or rather σ<sup>+</sup>) was investigated. Presence of ethanol greatly accelerates hydrolysis. The reaction mechanism is discussed.

C.O.C.

### Organic Pigments. III—Azo Compounds of 2-Naphthol

J. Lenoir *Peintures, pigments, vernis*, 36 (1960) 144-157  
*Chem. Abs.*, 54 (25 Aug 1960) 16863

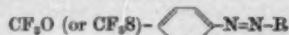
Extensive review including chemical structures and various commercial sources of supply of azo derivatives of 2-naphthol, e.g. Para Reds, Toluidine Reds, Dinitraniline Reds and Oranges, Lithol Reds, Reds from Lake C and other azo derivatives of sulphonated amines. C.O.C.

### Fluorine-containing Azo Dyes

L. M. Yagupolskii, B. M. Krasovitskii, V. A. Blinov, K. M. Sidneva, and D. G. Pereyaslova

*Ukrain. khim. zhur.*, 26, No. 3 (1960) 389-392

Synthesis and properties of azo dyes of formulae—



R = H-, R-, or e-acid, and of their fluorine-free analogues have been investigated. Fluorine causes considerable hypsochromic displacement of absorption max. but does not affect fastness of dyeings on wool. On the other hand, azoic-dyeings formed on cotton by coupling diazotized p-trifluoromethoxy- and p-trifluoromethylmercaptanilines with o-anisidine of β-hydroxynaphthoic acid, show marked increase in fastness to light compared with their non-fluorinated analogues, but an unchanged fastness to washing at the boil. G.J.K.

### Steric Effects in Di- and Tri-arylmethanes. II—Electronic Absorption Spectra of Derivatives of Michler's Hydrol Blue, Malachite Green, and Crystal Violet containing Hindered Dimethylamino groups

C. C. Barker, G. Hallas, and A. Stamp

*J.C.S.*, (Oct 1960) 3790-3800

Steric inhibition of the mesomerism of all or any of the dimethylamino groups in Crystal Violet (I) causes bathochromic shifts of the first frequency absorption band, whereas inhibition of the mesomerism of all of these groups in Malachite Green (II) and Michler's Hydrol Blue (III) causes small and moderate hypsochromic shifts, respectively. Inhibition of the mesomerism of one only of the dimethylamino groups in II or III causes bathochromic shifts of the first frequency band. The results now reported make it probable that the bathochromic shift observed in passing from Methylene Blue to Methylene Green should be ascribed to enforced rotation of the hindered dimethylamino group (cf. Dewar, *J.C.S.*, (1950) 2329; *Chem. Soc. Special Publ.*, No. 4 (1956) 79).

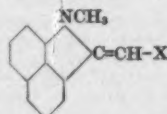
H.H.H.

### Derivatives of Benz(c,d)Indoline. V—Monomethincyanines

N. S. Dokunikhin and Ya. B. Shteinberg

*Zhur. obshch. khim.*, 30 (June 1960) 1989-1992

One symmetrical and four asymmetrical monomethincyanines of formula—



have been prepared and their absorption max. (visible range) measured: X = benzthiazolinium-, benzoxazolinium-, and quinolinium ethiodide; and 3,3-dimethylindoleninium methiodide. G.J.K.

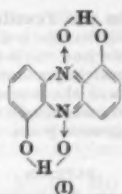
### Constitution of the Pigments of *Brevibacterium Crystalloiodinium* Sasaki, Yoshida et Sasaki

T. Irie, E. Kurosawa, and I. Nagaoka

*Bull. Chem. Soc. Japan*, 33 (Aug 1960) 1057-1059

The respective deep purple and yellow pigments from the culture medium of *Brevibacterium crystalloiodinium* were named by their discoverers (cf. title) crystalloiodinine A and B, owing to the resemblance of the antibiotic A to iodinine (cf. Clemons and McIlwain, *J.C.S.*, (1938) 479). A is now found to be identical with iodinine (1,6-dihydroxyphenazine di-N-oxide (I)), and when hydrogenated over Adams Pt catalyst gives 1,6-dihydroxyphenazine, identical with B.





H.H.H.

### Triazine Derivatives. II—Colour of Some Triazine Monoazo Dyes Containing Reactive Chlorine Atoms

V. I. Mur and G. N. Vorozhtsov

*Zhur. obshch. khim.*, 30 (June 1960) 1981–1985

Fourteen triazine monoazo dyes containing 1 or 2 reactive chlorine atoms in the ring, of formula—



have been investigated spectrophotometrically (visible range). They have been prepared by acylating *m*-phenylenediaminesulphonic acid with cyanuric chloride, diazotising and coupling with 1-phenyl-3-methyl-5-pyrazolone-4', 1-(2',5'-dichlorophenyl)-3-methyl-5-pyrazolone-4', and 1-(2'-chlorophenyl)-3-methyl-5-pyrazolone-5'-sulphonic acid. In five triazine dyes, R consisted of the copper complex of the azo compound. Change in pH (over the range 6–10), or substitution of one chlorine atom by a hydroxyl group, or by anilino-mono- or di-sulphonic acid, does not affect  $\lambda_{\max}$ . Substitution of both chlorine atoms by hydroxyl causes a hypsochromic displacement of  $\lambda_{\max}$ . G.J.K.

### Absorption Spectra of Methylene Blue adsorbed on Homolomic Bentonites suspended in Water

C. Sato

*Bull. Chem. Soc. Japan*, 33 (Aug 1960) 1148–1149

A previous observation of the changes in absorption spectra with time of Methylene Blue (I; C.I. 52015) adsorbed on montmorillonite (a phenomenon known as *metachromasy*), is now extended to the spectra of I adsorbed on homoionic bentonites suspended in water and having exchangeable cations of H, Na, K, NH<sub>4</sub>, and Ca. For the NH<sub>4</sub> and K cpd. the band at 570 m $\mu$  decreases in intensity remarkably, and the broad bands at 600 ~ 620 m $\mu$  and especially at 660 ~ 680 m $\mu$  increase in intensity markedly with time, while bands at 725 m $\mu$  and 770 m $\mu$  become apparent gradually (II). Changes in the H and Ca cpd. occur more slowly, and are scarcely observed in the strong band of the Na cpd. at 570 m $\mu$  and the weak one at 670 m $\mu$ . It is assumed that in the NH<sub>4</sub> and K cpd. a greater part of dye adsorbed initially in associated form on the outer surface of the clay particle migrates easily to the firm binding of the unassociated dye ion on the interlayer surface, but that this migration is impeded in the Ca and H cpd. and especially so in the Na cpd. No explanation can yet be offered for II. H.H.H.

### Infrared Absorption Spectra of Some Polycyclic Anthraquinone Vat Dyes

T. Urbanaki, W. Hofman and M. Witanowski

*Bull. acad. polon. sci., Sér. sci., Chim., géol. et géograph.*, 7 (1959) 215–221 (in English)*Chem. Abs.*, 54 (25 Aug 1960) 16834

Examination of the infrared absorption spectra in the 4500–750 cm<sup>-1</sup> range of dyes containing several condensed aromatic rings, the dyes being mixed with their own weight of nujol (cf. Crook and Taylor, *Chem. Abs.*, 52 (1958) 5976), revealed the following bands (*sh*, *w*, *s* and *b* = shoulder, weak, strong and broad, respectively): Cibacron Yellow GK (C.I. 61725)—3300*sh*, 3060*w*, 1655*s*, 1595, 1580*w*, 1560*w*, 1500*w*, 1345*s*, 1200*w*, 1040*w*, 980, 855*w* and 760*s*; Indanthren Golden Orange G (C.I. 59700)—3300*b*, 3080*w*, 1645*s*, 1600*a* (*sic*), 1580*w*, 1555*sh*, 1325*w*, 1310, 1290*sh*, 1155*w*, 1025*w*, 935*w*, 900*w*, 825*w*, 800*w* and 770*w*; Cibacron Blue BO—3300*w* and *b*, 3080*w*, 1635, 1600*sh*, 1585, 1555*sh*, 1350, 1300*w*, 1280*sh*, 1195*w*, 1160*w*, 1075*w*, 1035, 950*w*, 845*w*, 795*w* and 750; Sandothrene Yellow GN—3300*w* and *b*, 3060*w*, 1660*s*, 1595*s*, 1550*w*, 1485*sh*,

1345*w*, 1305*s*, 1270*s*, 1155, 1090*w*, 1060, 945, 900*w*, 860, 810*w* and 780*w*; Indanthren Blue RS (C.I. 69800)—3300*w* and *b*, 3085*w*, 1655, 1635*w*, 1585, 1490*s*, 1425*sh*, 1345*w*, 1310*w*, 1285–1270*s*, 1195*w*, 1155*w*, 1090*w*, 1060, 945, 900*w*, 860, 810*w* and 780*w*; Sandothrene Brilliant Orange NGK (similar to C.I. Vat Orange 19)—330*w* and *b*, 3080*w*, 1660*s*, 1635*sh*, 1595*sh*, 1575, 1550*sh*, 1495*sh*, 1415*w*, 1340*w*, 1320, 1280*w*, 1195*w*, 1155*w*, 1085*w*, 990*w*, 960*w*, 905*w*, 845*w*, 770*sh* and 760 cm<sup>-1</sup>. C.O.C.

### Study of the Coloured Metal-Hydroxyanthraquinone Compounds

A. K. Babko and T. N. Nazarchuk

*Raboty Khim. Rasvorov i Kompleks. Soedinenii*,*Akad. Nauk. Ukr. S.S.R.*, (2) (1959) 199–215*Chem. Abs.*, 54 (25 Aug 1960) 16249

Under conditions for colorimetric measurement, metal ions form definite compounds with Alizarin (C.I. 58000) (I) and Quinalizarin (C.I. 58500) (II). The following molecular ratios were established: Al:I = 1:1 and 1:2; Cu:I = 1:1, UO<sub>2</sub>:I = 1:1; Fe:I = 1:2; Sn(IV):I = 1:3, Zr:I = 1:1; Sn:II = 1:3. The Sn-quinalizarin compound is stable at pH 4–5. The Al-alizarin compound is stable at pH 7–10. The low value of the decomposition constant of Al alizarinate (10<sup>-18</sup>) indicates that in the metal alizarinate the metal ions replace the H atoms of both phenol groups. C.O.C.

### Naphthoquinone Dyes in Aqueous Dispersion, Analogues of Anthraquinone Dyes

M. F. Sartori

*Ann. chim. (Rome)*, 49, 2157–2163*Chem. Abs.*, 54 (25 Aug 1960) 16834

Derivatives of 2,3-dimethyl-1,4-naphthoquinone-I were prepared and their dyeing properties on polyethylene terephthalate fibres compared with those of the corresponding anthraquinone dyes. The dyebaths were set with 0.1 g dye in 500 ml water at 82°C. The sample was entered, and boiled for 1 h, washed with hot water, then in 0.7% aq. alkyl sulphate, again in hot water and dried. The results were as follows (dye,  $\lambda_{\max}$  in m $\mu$ , ( $\epsilon \times 10^{-3}$ ), % exhaustion, % penetration): 5-amino-1, 475 (3-7), 64, 60; benzoyl-1, 425 (4-6), 73, 65; 5-amino-6,8-dibromo-2,3-dimethyl-1,4-naphthoquinone 480 (5-5), 30, 75; 5-amino-6-bromo-8-hydroxy-2,3-dimethyl-1,4-naphthoquinone, 560 (7-3), 50, 50; 6-bromo-5,8-diamino-2,3-dimethyl-1,4-naphthoquinone, 575–620 (7-8-6-6), 40, 65; 8-anilino analogue of the preceding dye, 630 (10-8), 34, 50. Preparation of these dyes is described in detail. C.O.C.

### $n-\pi^*$ Transition Band of Acenaphthenequinone

A. Kuboyama

*Bull. Chem. Soc. Japan*, 33 (Aug 1960) 1027–1030

The weak absorption band ( $\alpha$ -band) ( $\epsilon_{\max} \approx 20$ ) near 500 m $\mu$  of acenaphthenequinone (I) is measured in benzene, chloroform, dioxan, carbon disulphide, and diethyl ether, and identified as the  $n-\pi^*$  transition band on the basis of the observed results on intensity and solvent effect. In dioxan, the  $n-\pi^*$  transition bands of the o-quinones, including the  $\alpha$ -band (as shown for I,  $\beta$ -naphthoquinone, and phenanthraquinone), largely shift towards shorter wavelengths, and this effect, which appears to be due to formation of molecular complexes, is discussed qualitatively. The relative height of the two main peaks of the  $\alpha$ -band of I depends markedly upon solvents, and these two peaks are assumed to be due to the two  $n-\pi^*$  transitions whose transition energy differences is ca. 700 cm<sup>-1</sup>. H.H.H.

### Thioindigo Dyes. V—Effect of Methyl Groups and Halogen on the Colour of Thioindigo

N. S. Dokunikhin and Yu. E. Gerasimenko

*Zhur. obshch. khim.*, 30 (June 1960) 1987–1989

Absorption max. (in benzene) of 5,5'-(I) and 7,7'-(II) dimethyl- and dihalogeno- (Cl, Br, I) substituted thioindigo show for I a weaker bathochromic effect in the case of dimethyl, whilst for II a stronger effect. The preparation of *S*-(*o*-iodophenyl)-thioglycolic acid, 7-iodo-3-oxothionaphthene, and 7,7'-diiodothioindigo is described. G.J.K.

### Some Substituted Derivatives of Copper Phthalocyanine (C.I. 74160)

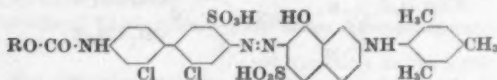
D. Colafitis

*Compt. rend.*, 250 (1960) 328–330*Chem. Abs.*, 54 (25 Aug 1960) 16464

The tetramethyl, tetrakis(chloromethyl), tetrakis(hydroxymethyl) and octamethyl derivatives of Cu



(R = Alk of > 5C, alkoxyethyl, or phenoxyethyl; X = CH<sub>3</sub> or Cl) are diazotised and coupled with  $\gamma$  acid or its amides or *N*-aryl derivatives to give red acid dyes, which exhaust from neutral or weakly acid baths. Good wet fastness is given by compounds of mol.wt. > 600. Thus 2,2'-dichlorobenzidine-5-sulphonic acid is acylated with *n*-octyl chloroformate and the product is diazotised and coupled with *N*-2,4,6-trimethylphenyl- $\gamma$  acid to give—

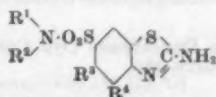


(R = C<sub>8</sub>H<sub>17</sub>) which is a violet-red.

E.S.

#### Red and Violet Disperse Monoazo Dyes from 2-Aminobenzothiazole-6-sulphonamides

S BP 851,910 (Switzerland 13 June 1957)  
The title dyes are obtained by diazotising 2-amino-benzothiazole-6-sulphonamides—

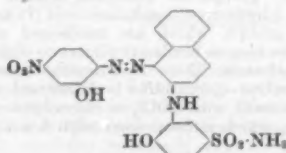


(R<sup>1</sup> = H, Alk, hydroxy- or cyano-alkyl, alkenyl, or alkoxyalkyl; R<sup>2</sup> = H, Alk, hydroxyalkyl, alkoxyalkyl, Ar, cycloalkyl, aralkyl, or hydroxyaralkyl; R<sup>3</sup>, R<sup>4</sup> = H, Alk, or together = CH-CH-CH-CH) and coupling with suitable derivatives of aniline containing *N*-alkyl, -hydroxyalkyl, -cyanoalkyl, etc. groups. Thus 2-amino-benzothiazole-6-sulphonamethylamide is diazotised by dissolving in 85% phosphoric acid, cooling to 5°C and adding NaNO<sub>2</sub>. After acetic acid is added, coupling with *N*-cyanoethyl-*N*-hydroxyethylamine gives a bright red for cellulose acetate.

E.S.

#### Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool

FBY BP 852,262 (Germany 7 Aug 1958)  
*o*-Aminophenol, *o*-anisidine, anthranilic acid, and their derivatives containing substituents such as Cl, NO<sub>2</sub>, SO<sub>2</sub>CH<sub>3</sub>, SO<sub>2</sub>NH<sub>2</sub>, etc. are diazotised and coupled with *N*-*o*-hydroxyphenyl-2-naphthylamines, which may also contain substituents, and the resulting monoazo compounds are converted into 1:2 Cr- or Co-complexes, which dye wool grey, green, or olive from neutral baths. Thus 3-hydroxy-2-naphthoic acid is heated with 2-amino-phenol-4-sulphonamide in presence of NaHSO<sub>3</sub> until evolution of CO<sub>2</sub> ceases, to give 2-(2'-hydroxyphenyl)-naphthylamine-5-sulphonamide, which is coupled with diazotised 2-amino-5-nitrophenol in presence of dimethyl formamide, giving—

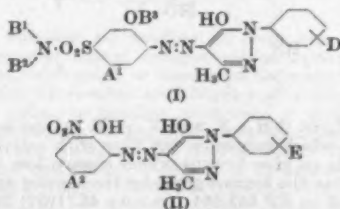


Heating in dimethyl formamide with Cr(OH)<sub>3</sub> and 80% formic acid gives the complex, which dyes wool greenish khaki.

E.S.

#### Red Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool and Nylon

BASF BP 852,363 (Germany 15 Dec 1956)  
Mixed complexes derived from 1 atom of Cr and 1 mol. of each of 2 monoazo pyrazolones—



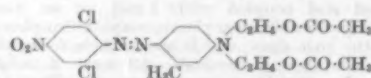
(A<sup>1</sup>, A<sup>2</sup> = Hal or Alk of < 5 C; B<sup>1</sup>, B<sup>2</sup>, B<sup>3</sup> = H or Alk of < 5 C; D = H, Hal, or Alk of < 5 C; E = H or sulphonyl) give bluer reds than similar complexes from 2 mol. of I, better solubility, cotton reserve, and fastness to rubbing than those from 2 mol. of II in which D = H, and are more level-dyeing and faster to moisture than those from 2 mol. of II in which D = sulphonyl. Thus the complex from 1 atom of Cr and 1 mol. of the monoazo compound 5-amino-4-methoxytoluene-2-sulphonamide-3-methyl-1-phenyl-5-pyrazolone is heated with the monoazo compound 5-amino-4-hydroxy-3-nitrotoluene-3-methyl-1-phenyl-5-pyrazolone in presence of salicylic acid to give the mixed complex which dyes wool red from a neutral or acetic acid bath.

E.S.

#### Monoazo Disperse Dyes from *O*-Acylated *N*-Hydroxyalkylarylamines

ICI BP 852,396 (17 Sept 1958)

*O*-Acyl derivatives of *NN*-bis(hydroxyalkyl)arylamines are coupled with diazotised 4-nitroaniline or its 2- or 6-halogen derivatives to give red and brown disperse dyes, faster to sublimation than the corresponding dyes without *O*-acyl groups. Thus diazotised 2,6-dichloro-4-nitroaniline coupled with *NN*-bis(β-acetoxyethyl)-*m*-toluidine gives—



which dyes polyester fibres reddish brown.

BP 852,493 (2 July 1958)

Red and violet disperse dyes are given by coupling the above-mentioned coupling components with diazotised 2-cyano-4-nitroaniline (I) or its 6-halogeno derivatives. Thus diazotised I coupled with the dibenzoic ester of *NN*-bis(β-hydroxyethyl)-*m*-toluidine gives a bluish red of good fastness to dry heat and steam pleating on polyester fibres.

E.S.

#### Yellow Monoazo Pyrazolone Disperse Dyes Containing Thiocyanate Groups

FBY BP 852,400 (Germany 25 Nov 1957)

The title dyes are obtained by coupling diazo compounds of the benzene series with suitable pyrazolones, thiocyanate groups being present in either or both components, and SO<sub>2</sub>H and COOH groups being absent. Thus the monoazo compound *p*-thiocyanatoaniline-3-methyl-1-phenyl-5-pyrazolone gives almost the same yellow on cellulose acetate, nylon, and polyester fibres.

E.S.

#### Fatty Acid Salts of Metal(Chromium)-complex Monoazo Dyes—Wood Stains

ICI BP 851,558 (2 May 1958)

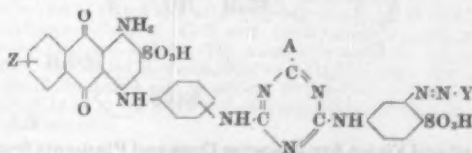
The 1:1 Cr-complexes of suitable *oo'*-dihydroxy (or *o*-carboxy-*o'*-hydroxy)monoazo dyes are heated with a long-chain fatty acid (especially oleic acid) to give complexes of value as wood stains because of their ready solubility in hydrocarbons and their fastness to light and to bleeding into cellulose nitrate and shellac finishes. Or the metal-free dye may be heated with e.g. oleic acid and hydrated CrCl<sub>3</sub>. Thus the monoazo compound anthranilic acid-3-methyl-1-phenyl-5-pyrazolone treated in this way gives a yellow.

E.S.

#### Green Anthraquinone Triazine Monoazo Acid Dyes and Reactive Dyes for Cellulose

Ciba BP 849,115 (Switzerland 27 Jan 1956)

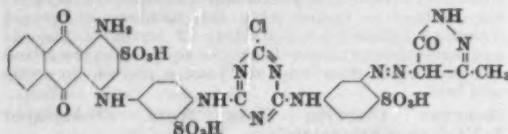
Monoazo compounds of the type—



(Z = H or SO<sub>2</sub>H; A = Cl or a substituent of < 13 C, not having the character of a dye, and preferably bound to the triazine ring through an N atom; Y = residue of barbituric acid or of a 3-methyl-5-pyrazolone) are acid dyes if < 3 SO<sub>2</sub>H groups are present, and reactive dyes for cellulose if A = Cl and > 2 SO<sub>2</sub>H groups are present. They



are faster to light than the similar dyes disclosed in BP 466,886 (J.S.D.C., 53 (1937) 406). Thus 1-amino-4-(4'-aminophenylamino)-anthraquinone-2,3'-disulphonic acid is condensed with 1 mol. of cyanuric chloride, and the product is then condensed with 1 mol. of the amino-monoazo compound formed by removal of the acetyl group from the monoazo compound 3-aminoacetanilide-4-sulphonic acid→3-methyl-5-pyrazolone, giving

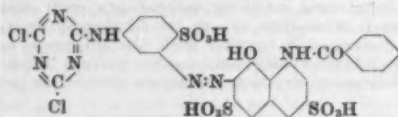


which dyes cellulose bluish green by a pad-dyeing process followed by fixation with alkali. E.S.

#### Reactive Monoazo Dyes for Cellulose having a Dihalogenotriazine Residue

Ciba BP 851,537 (Switzerland 20 June 1956)

One mol. of a cyanuric halide is condensed with 1 mol. of an aromatic primary diamine, and the product is then diazotised and coupled with 1 mol. of an *o*-coupling naphthol (preferably an acylaminonaphtholsulphonic acid) to give the title dyes, which are suitable for application by the cold-dyeing method. At least 2 solubilising ( $\text{SO}_3\text{H}$  and/or  $\text{COOH}$ ) groups are present. Thus cyanuric chloride is condensed with 1 mol. of *m*-phenylenediamine-4-sulphonic acid and the product is diazotised and coupled with *N*-benzoyl-*H* acid to give—

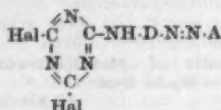


which dyes cellulose bluish red at 25–35°C from a bath containing salt and  $\text{Na}_2\text{CO}_3$ . E.S.

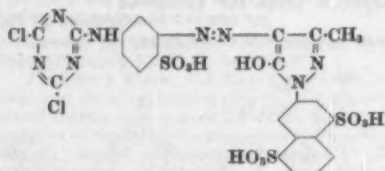
#### Yellow Monoazo Reactive Dyes for Cellulose

Ciba BP 852,120 (Switzerland 29 June 1956)

Dyes of type—



(A = residue of *o*-coupling keto-methylene compound; D = residue of a diazo-component; A or D or both of them contain a bicyclic aromatic nucleus; at least 2  $\text{SO}_3\text{H}$  or  $\text{COOH}$  groups are present) are suitable for application by the cold-dyeing process from an alkaline bath. Thus 1 mol. of cyanuric chloride is condensed with 1 mol. of *m*-phenylenediamine-4-sulphonic acid, and the product is diazotised and coupled in presence of  $\text{NaHCO}_3$  with 3-methyl-1-naphthyl(2')-5-pyrazolone-4',8'-disulphonic acid to give—

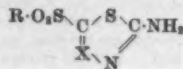


E.S.

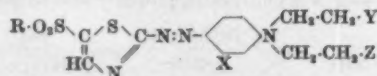
#### Red and Violet Azo Disperse Dyes and Pigments from Heterocyclic Diazo Components

S BP 851,909 (Switzerland 29 May 1957)

Heterocyclic amines—



(X = N, CH, C-Alk, C- $\text{CF}_3$ , C-phenyl, C-(methylphenyl), C-(ethylphenyl), C-(dimethylphenyl) or C-(halogenophenyl); R = Alk or alkenyl of 1–4 C which may carry Hal, OH, or CN substituents, or  $\text{NH}_2$  which may carry 1 or 2 Alk, Ar, aralkyl or cycloalkyl radicals) are diazotised and coupled with suitable *NN*-disubstituted arylamines to give the title dyes. The compounds—

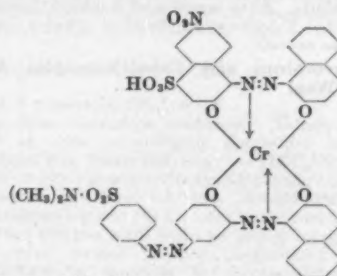


(for R see above; X = H, Cl, or  $\text{CH}_3$ ; Y = H,  $\text{CH}_3$ ,  $\text{C}_2\text{H}_5$ , OH, acetoxy, or  $\text{O-CO-NHC}_2\text{H}_5$ ; Z = CN, acetoxy, or  $\text{O-CO-NHC}_2\text{H}_5$ ) are especially interesting. Thus the monoazo compound 2-amino-6-methylsulphonylthiazole→*NN*-bis(acetoxyethyl)-*m*-toluidine dyes polyester and cellulose acetate fibres bright red, and the monoazo compound in which R =  $\text{CH}_3$ , X = Cl; Y = Z =  $\text{O-CO-NHC}_2\text{H}_5$ , colours cellulose acetate spinning solution bright red. E.S.

#### Metal-Monoazo Dye-Disazo Dye Complexes—Dyes for Wool, Lacquers, etc.

Gy BP 851,861 (Switzerland 26 Jan 1956)

The title products contain 1 atom of a metal, preferably Cr, combined with 1 mol. of a metallisable monoazo compound and 1 mol. of a metallisable disazo compound, and those intended for dyeing wool contain a sulphonyl group and > 2  $\text{SO}_3\text{H}$  groups. Thus the 1:1 Cr-complex of the monoazo dye 1-amino-6-nitro-2-naphthol-4-sulphonic acid→β-naphthol is boiled in presence of aq. NaOH with the disazo dye 3-amino-4-hydroxyazobenzene-4-sulphondimethylamide→β-naphthol to give—



which dyes wool black.

E.S.

#### Disazo Pigments having Pyreneamide Groups

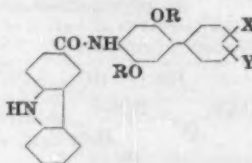
Ciba BP 851,740 (Switzerland 7 Sept 1956)

The  $\text{COOH}$  groups of insoluble disazo compounds derived from 3-hydroxy-2-naphthoic acid (I) are converted into  $\text{COHal}$  groups, which are condensed with aminopyrenes to give blue to bordeaux pigments of good fastness to light and solvents. Thus the aminomonoazo compound 2,5-dichloroaniline→*p*-cresidine is diazotised and coupled with I. Treatment with  $\text{SOCl}_2$  in chlorobenzene gives the acid chloride, which is condensed with 3-aminopyrene to give a violet. E.S.

#### Arylides of 2-Hydroxycarbazole-3-carboxylic Acid—Coupling Components for Azoic Browns

FBy BP 851,538 (Germany 11 July 1956)

4-Amino-2,5-dialkoxydiphenyls are condensed with 2-hydroxycarbazole-3-carboxylic acid (I) to give arylides—



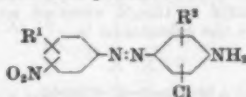
(R =  $\text{CH}_3$  or  $\text{C}_2\text{H}_5$ ; X and Y = H or non-ionic substituents) which couple on the fibre with suitable diazo compounds to give browns which change less on after-soaping than the browns given by the simpler arylides of I described in BP 343,164 (J.S.D.C., 48 (1932) 21). Thus



diazotised *p*-anisidine is added to an acid solution of *p*-benzoquinone, when nitrogen is evolved and 4'-methoxy-diphenylquinone-2,5 is obtained. This is reduced and methylated to give 2,5,4'-trimethoxydiphenyl, which is then nitrated and reduced, giving the 4-amino compound. Condensation with I in toluene with  $\text{PCl}_5$  gives the arylide. Cotton impregnated with an alkaline solution of it and developed in a solution of diazotised *m*-chloroaniline is dyed yellowish brown. E.S.

#### Stabilised Diazo Compounds from Aminomonoazo Compounds

FH BP 851,858 (Germany 20 Dec 1955)  
The diazonium chlorides, sulphates, borofluorides and the diazonium chloride-zinc chloride double salts derived from aminomonoazo compounds—



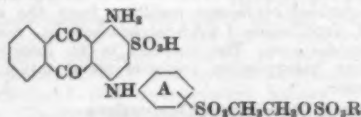
( $\text{R}^1 = \text{H, Hal, Alk, or OAlk}$ ;  $\text{R}^2 = \text{H, Alk, or OAlk}$ ) have good stability in the solid state, largely due to the presence of the Cl atom. Thus the aminomonoazo compound obtained by coupling diazotised *p*-nitroaniline with *m*-chloroaniline-*N*-methane sulphonic acid and splitting off the methanesulphonic acid group by hydrolysis, is diazotised and  $\text{ZnCl}_2$  added to give the double salt, which is salted out, filtered off and dried at a moderate temperature. E.S.

#### Red Azoic Dye and Pigment

FH BP 852,589 (19 July 1958)  
The monoazo compound made in substance or on the fibre by coupling diazotised 3-amino-4-methoxybenzaniline (I) with the 4-chloro-2,5-dimethoxyaniline of 3-hydroxy-2-naphthoic acid (II) is faster to solvents and to bleeding into undyed polyvinyl chloride than the similar dye described in BP 733,160 (J.S.P.C., 71 (1955) 677) prepared from diazotised I and the 5-chloro-2,4-dimethoxyaniline of II, and on the fibre is faster to washing and peroxide than the dye from diazotised 3-amino-4-methoxybenzamide and the 4-chloro-2,5-dimethoxyaniline of II. E.S.

#### Water-soluble Anthraquinone Dyes for Wool and Cellulose

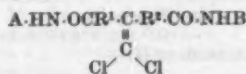
ICI BP 852,604 (25 July 1958)  
Reddish blue dyes of excellent washing fastness have formula—



$\text{R} = \text{Alk, subst. or unsubst. Ar; A may contain Cl, Br, Alk, or alkoxy}$ . They are obtained from analogous  $\beta$ -hydroxyethylsulphonates (BP 401,132) by condensation with organic sulphonic acid halides. Thus, 1-amino-4-(3'- $\beta$ -hydroxyethylsulphonyl) anilinoanthraquinone in pyridine with methane sulphonyl chloride gives (A is unsubst.,  $\text{R} = \text{CH}_3$ ) a bright reddish blue dye having excellent fastness to washing and milling on wool and to washing and light on cotton. A.T.P.

#### Anthraquinone Vat Dyes and Pigments

Gy BP 851,557 (Switzerland 4 May 1957)  
Dyes of formula—

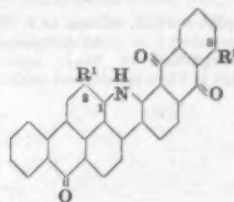


(A, B = vatable AQ residues, at least one being a 1-anthraquinonyl radical unsubst. in the 4-position but possibly otherwise subst. or fused with heterocyclic rings, and especially where subst. are halogen and/or aroylamino;  $\text{R}^1, \text{R}^2 = \text{subst. or unsubst. phenylene or diphenylene radicals}$ ) are vat dyes or pigments of excellent fastness to light, chlorine, washing and soda boiling. They are obtained by condensing 1 mol. of a dicarboxylic acid  $\text{HOOC-R}^1\text{-C(=CCl}_2\text{)-R}^2\text{-COOH}$  (or  $\text{HOOC-R}^1\text{-CH(CCl}_2\text{)-R}^2\text{-COOH}$ , followed by HCl elimination as below) or its

derivatives with 2 mol. of the same or different anthraquinone compound, preferably in two stages where  $\text{A} \neq \text{B}$ , or by condensing aroylamino anthraquinones containing phenylene or diphenylene radicals in the aroyl groups with trichloroacetaldehyde, followed by HCl elimination by, e.g., heating in pyridine in presence of NaOH. Preferred are the "hot-dyeing" dyes derived from dichloroethylene bis-diphenylcarboxylic acid. Thus, the yellowish red vat dye where  $\text{A} = \text{B} = 1\text{-amino-6,7-dichloroanthraquinonyl}$ ,  $\text{R}^1 = \text{R}^2 = \text{diphenylene}$ . A.T.P.

#### Anthraquinone Vat Dyes

FH BP 852,517 (Germany 26 Nov 1955)  
Vat dyes of excellent light fastness and good fastness to washing and soda boiling are of formula—



( $\text{R}^1 = \text{Alk or Ar; R}^2 = \text{NH}_2 \text{ or acylamino}$ ). They are obtained from Bz-1-halogenbenzanthrone subst. at Bz-2 by Alk or Ar by reaction with a 1,5-diaminoanthraquinone or *N*-monoacyl derivatives to give benzanthrone-amino anthraquinones, which are finally cyclised by melting with an alkaline condensing agent. Where an acylamino group is present in the 5-position, cyclisation is effected by the method of BP 339,396 (Examples 7,8) to prevent hydrolysis. A.T.P.

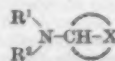
#### Salts of Sulphuric Acid Esters of Leuco Vat Dyes

DE BP 852,518 (Switzerland 25 Oct 1955)

The title cpd. are obtained from leuco anthraquinone vat dyes which are difficult to esterify, e.g. anthrimides, anthraquinone-carbazoles, 1-arylaminoanthraquinones containing acylamino, alkoxy or halogen in the 4, 5, or 8 positions, and bis-arylaminoanthraquinones, by treatment in a mixture of pyridine and/or homologues or mixtures (I) with one or more tertiary amines (II) (10-60%) containing a  $\text{N-CH}$  group in which (i) there are bound to the N atom either two saturated hydrocarbon radicals together containing 3-11 C or a single saturated divalent hydrocarbon radical forming a ring with the N atom, each hydrocarbon radical having  $\geq 7$  C, and (ii) there are bound to the C atom, two saturated hydrocarbon radicals together containing  $\geq 8$  C atoms where the N atom forms part of a ring, a single saturated divalent hydrocarbon radical forming, together with the C atom, a carbon ring, each hydrocarbon radical having  $\geq 6$  C, or (iii) there are bound to the C atom a  $\text{CH}_2$  group, and to the N atom a saturated hydrocarbon radical of  $\geq 7$  C, and the C and N atoms, together with a saturated divalent hydrocarbon radical, form a ring, e.g. methyl di-*iso*-propylamine, *N*-*s*-butyl-piperidine, *N*-methyl-2,6-dimethylpiperidine, *N*-cyclohexylpiperidine. In presence of finely divided Fe, Co, Zn, Cu, or Cu alloys, and with  $\text{ClSO}_3\text{H}$  in the form of its addition products with I, are formed the sulphuric acid esters, finally converted to the title compounds, especially the Li, Na, K,  $\text{NH}_4$ , or hydroxyalkylamine salts. Thus  $\text{ClSO}_3\text{H}$  (20) is added with cooling to  $\alpha$ -picoline (160). Fe powder (25) is added and after stirring 15 min at 18-20°C, diethylisopropylamine (41.5) and 1,5-dibenzamidoanthraquinone (10) are added. The mass is heated for 5 h at 55-57°C and added to water (600) and  $\text{Na}_2\text{CO}_3$  (36). Bases are distilled off and salt is added, 83% of the Na salt of the  $\text{H}_2\text{SO}_4$  ester of leuco 1,5-dibenzamidoanthraquinone being obtained.

BP 852,519 (Switzerland 25 Oct 1955)

Similar claims to the above, where II are of formula—



(X = subst. or unsubst. pentamethylene;  $\text{R}^1 = \text{Alk}$  of 1-4 C,  $\text{R}^2 = \text{Alk or cyclohexyl}$ ). The products are at least

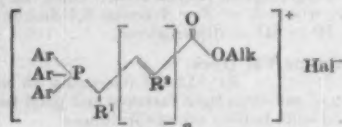
as good as those resulting from II = dimethylcyclohexylamine; BP 719,621 and BP 745,365 (J.S.D.C., 71 (1955) 157; 72 (1956) 394). A.T.P.

#### Polyene Carboxylic Acids and Esters—Food Dyes

F. Hoffmann-La Roche & Co.

BP 850,137 (Switzerland 27 Nov 1957)

A phosphonium halide of formula—



( $n = 0, 1, 2, \text{ or } 3$ ;  $\text{R}^1 = \text{H}$  or, when  $n = 0$ ,  $\text{H}$  or  $\text{CH}_3$ ;  $\text{R}^2 = \text{CH}_3$  and  $\text{H}$  alternately) is dehydrohalogenated with a metal-organic compound or basic agent to yield a phosphorane. This is then condensed with an aldehyde of formula—



(broken line = an optional bond;  $\text{R}^3 = \text{CH}_3$  and  $\text{H}$  alternately;  $m = 2, 4, \text{ or } 6$  when  $\text{R}^1 = \text{CH}_3$  and  $1, 3, \text{ or } 5$  when  $\text{R}^1 = \text{H}$ ;  $m + n < 7$ ). Triarylphosphine oxide is split off from the condensate (by heating, if desired), the product partly hydrogenated at the triple bond and, if necessary, isomerised and then, if desired, hydrolysed. Thus methoxycarbonyl methyltrimethylphosphonium bromide dissolved in anhydrous methylene chloride was treated in an atmosphere of  $\text{N}_2$  with 2*N*-methanolic sodium methylate solution. 2,7,11-Trimethyl-13-[2',6',6'-trimethylcyclohexen-(1')-yl]-tridecapentaen-(2,6,8,10,12)-yn-(4)-al-(1) in methylene chloride was then added and the mixture refluxed for 5 h. This yielded orange crystals of 4,9,13-trimethyl-15-[2',6',6'-trimethylcyclohexen-(1')-yl]-pentadecaheptaen-(2,4,8,10,12,14)-yn-(6)-oic-(1)-acid methyl ester. This on reduction gave 6,7-mono-*cis*-4,9,13-trimethyl-15-[2',6',6'-trimethylcyclohexen-(1')-yl]-pentadecaheptaen-(2,4,6,8,10,12,14)-oic-(1)-acid methyl ester which when refluxed with petroleum ether under  $\text{CO}_2$  yielded orange red crystals of all-*trans* 4,9,13-trimethyl-15-[2',6',6'-trimethylcyclohexen-(1')-yl]-pentadecaheptaen-(2,4,6,8,10,12,14)-oic-(1)-acid methyl ester. This on hydrolysis yields crystals of the free acid which has absorption maximum at 430 mμ,  $E_{1\%}^{1\text{cm}} = 2235$ , in petroleum ether. C.O.C.

#### C.I. 61135 as Bluing Agent in Laundry Detergents

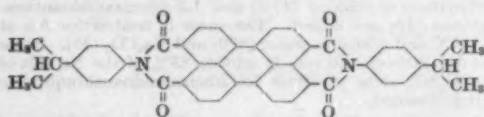
Thomas Hedley & Co. BP 850,531 (U.S.A. 1 Oct 1956)

The water-soluble alkali-metal salts of the condensate of 1-amino-4-bromo-2-anthraquinone sulphonic acid (2 mol) and *pp'*-diaminodiphenyldimethylmethane are excellent bluing agents for use in laundry detergents. The Na salt, C.I. 61135, is preferred. C.O.C.

#### Dye from Perylene Tetracarboxylic Acid

FH BP 852,202 (Germany 31 Mar 1956)

A red dye of good clarity and fastness in printing inks, lacquers and plastics is obtained by condensing perylene-3,4,9,10-tetracarboxylic acid dianhydride with *p*-isopropylaniline, e.g. in conc. HCl at 210–220°C for 12 h.



A.T.P.

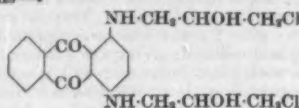
#### Blue Disperse Dyes for Polyamide and Polyacrylonitrile Fibres

ICI

BP 850,977 (4 Oct 1957)

Water-insoluble anthraquinones containing at least one halogenoalkoxy or halogenoalkylamino group (I) dye nylon and acrylic fibres in a very high degree of washing fastness. I are attached directly to a C atom of the anthraquinone

nucleus or through an alkoxy, alkylamino or arylamino group. Thus—



dyes nylon blue, very fast to washing.

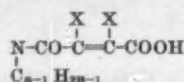
A.T.P.

#### Halogenoacyl Dyes for Wool and Cellulosic Fibres

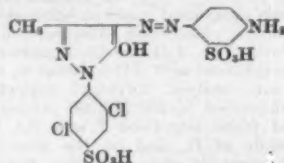
Ciba

BP 848,742 (Switzerland 27 Sept 1957)

Water-soluble dyes containing at least one water-solubilising group, e.g. sulphonamide, sulphone, and especially  $\text{COOH}$ ,  $\text{SO}_3\text{H}$ , and the residue of a chlorinated or brominated maleic or fumaric acid bound through one of its  $\text{CO}$ -groups to the N atom of an  $-\text{NH}_2$  group especially where the substituent is—



(one X = Cl, other is H;  $n$  is preferably 1) dye wool and cellulosic fibres in bright colours of good light and outstanding washing fastness. They are obtained from suitable dyes or intermediates (e.g. diazo or coupling components) containing acylatable amino groups by acylating with a halide of chlorinated or brominated maleic or fumaric acid and preferably with bromo- or chloromaleic anhydride. Thus, 1-amino-4-*p*-amino-anilinoanthraquinone-2-sulphonic acid and



with chloromaleic anhydride give, respectively, greenish blue and golden yellow dyes for cotton, of excellent washing fastness. A.T.P.

#### Naphthoylene Diarylimidazol Vat Dyes

General Aniline

BP 848,768 (U.S.A. 26 June 1957)

Reddish brown vat dyes are obtained by chlorinating or brominating at 60–80°C for 20–40 h an aq. dispersion of the separated *cis*-isomer resulting from the condensation of naphthalene-1,4,5,8-tetracarboxylic acid and *o*-phenylenediamine. The method is an improvement over known halogenation methods using solvents, e.g. nitrobenzene. A.T.P.

#### Halogenated Fluorescein Derivatives

Coty

BP 852,057 (U.S.A. 1 May 1958)

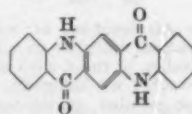
Commercial chlorinated and/or brominated fluorescein compounds (I) are treated with excess of acylating agent in the cold in presence of a basic, e.g. pyridine, or an acid, e.g.  $\text{H}_2\text{SO}_4$ , catalyst. Filtering, washing the acylated compound with a suitable solvent to remove catalyst and impurities, e.g.  $\text{CH}_3\text{OH}$ , converting to an alkali-metal salt derivative by a suitable reducing agent (alkali-metal hydride or alcoholate) and acidifying with a non-oxidising acid having at least the ionic strength of acetic acid gives a purified I. The new products are free from components responsible for skin irritation when used in lipsticks and cosmetics. A.T.P.

#### Linear Quinacridone Pigments

DuP

BP 851,976 (U.S.A. 22 Nov 1957)

The linear quinacridone (I)—



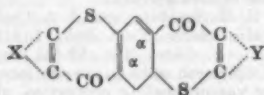
in the  $\gamma$ -crystal phase (see BP 828,052; J.S.D.C., 76 (May 1960) 318) and having a surface area of  $< 60 \text{ m}^2/\text{g}$  is obtained by oxidising the corresponding 6,13-dihydroquinacridone and treating the crude product with dimethyl

formamide to convert it into the required crystal phase. It can be converted into rosinated colour lakes containing 50–80% of I, advantageously combined with a sub-stratum of the Ca salt of hydrogenated rosin. The pigment is especially useful where depth of mass tone and transparency obtained usually by the specified particle size are not desirable, e.g. brilliant red automotive enamels. A.T.P.

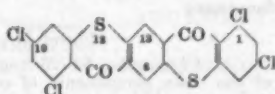
#### Thiachromone Pigments

ICI BP 851,571 (30 July 1958)

Bright yellow to purple pigments of good fastness to light, heat and solvents, suitable for paints, printing inks, rubber, synthetic polymers, are *lin*-benzobisthiachromones—



(X, Y = atoms necessary to complete a benzene ring or a system of fused 6- or 6- and 5-membered rings, and where the rings may be non-ionically subst.). They are obtained by fusing the requisite dicarboxylic acid (COOH at  $\alpha,\alpha$  C atoms) (I) by heating with dehydrating agents. I may be obtained from 2,5-dihalogenoterephthalic esters and thiophenols, thionaphthols or thiol deriv. of carbocyclic or heterocyclic epd., followed by hydrolysis of the ester group. Where the rings formed by X, Y contain as substituents ureido, thioureido, benzylideneamino or acylamino, the pigments may be obtained by treating corresponding amino compounds with an isocyanate, isothiocyanate, aldehyde or a functional derivative of an organic acid. Where X, Y form halogeno-subst. benzene rings, the pigments are bright yellow, e.g. 1,3,8,10-tetrachloro-*lin*-benzobisthiachromone—



has excellent fastness to out-door exposure in paints. Where X, Y form acylamino subst. benzene rings, the pigments are bright red and the 2,9-di(4'-chlorbenzamido)-compound is especially useful for mass coloration of cellulose acetate and polystyrene. A.T.P.

#### Water-soluble Phthalocyanine Dyes

BASF BP 848,782 (5 Mar 1959)

Phthalocyanines (I) containing at least one hydroxy-alkylaminomethyl group (II) (or a mixture of) are sulphated at preferably 10–30°C. II are obtained from halogenomethylated-I and hydroxyalkylamine or from aminomethyl-I and hydroxyalkyl halides or alkylene oxides. The dyes have, in addition to the known fastness of this class of dyes, excellent fastness to washing and kier-boiling. Especially useful are dyes from tris- and/or tetrakis-*N,N*-di-( $\beta$ -hydroxyethylaminomethyl)-Cu-phthalocyanines by sulphating, e.g. in 10 pt. 100% H<sub>2</sub>SO<sub>4</sub> at 0–4°C, adding to aq. NH<sub>3</sub> and salting out. A.T.P.

#### Non-flocculating Phthalocyanine Pigment Compositions

BASF BP 851,494 (Germany 24 Nov 1956)

Phthalocyanine pigment compositions (I) showing decreased flocculation in presence of organic solvents contain  $> 25\%$  of vanadium phthalocyanine (II). Because of inferior brilliance of II, 2–5% is preferred and is best incorporated by dissolving with the main pigment in conc. H<sub>2</sub>SO<sub>4</sub> and drowning into water. I may be used for stable lacquers, e.g. nitrocellulose or alkyd resin lacquers, and in printing inks. A.T.P.

#### Halogenated Phthalocyanine Pigments

ICI BP 850,237 (31 Mar 1958)

Cu polychloro-bromophthalocyanines containing 14.5–16 Hal (0.5–4 Br) atoms/mol. are yellower than, and of equal brightness and tinctorial strength (and thus more economical) to known polychlorophthalocyanines. They are obtained by treating hot Cu phthalocyanine (C.I. 74160) (I) with Cl<sub>2</sub> and adding between 8–100 parts Br<sub>2</sub>/100 I before 40% of the total Cl<sub>2</sub> has been used. Thus I (94) is added to a melt of AlCl<sub>3</sub> (394) and NaCl (82.5) at

155–165°C. Br<sub>2</sub> (19.6) is added over 15 min and Cl<sub>2</sub> (214) passed in over 15 h at 185–190°C. The mass is drowned into H<sub>2</sub>O (4000) and 35% HCl (100) to give (optionally H<sub>2</sub>SO<sub>4</sub> purified) a bright green pigment. A.T.P.

#### Phthalocyanine Sulphur Dyes

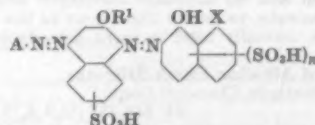
CFM BP 848,880 (Germany 10 May 1957)

Phthalocyanines or their metal derivatives react with ClSO<sub>3</sub>H and hydroxylamine or its salts or primary nitroparaffins of chain length  $> 4$  C atoms in presence of compounds of elements of Periodic Groups Va or VIA, e.g. NH<sub>3</sub> molybdate, V<sub>2</sub>O<sub>5</sub>, at 70–150°C to give polyaminophthalocyanines (I). Further reaction, without isolation, with  $< 5$  mol. S<sub>8</sub>Cl<sub>2</sub>/mol. I gives greenish to bluish-olive grey dyes of excellent light fastness (cf. BP 816,656 (J.S.D.O., 75 (1959) 561)). A.T.P.

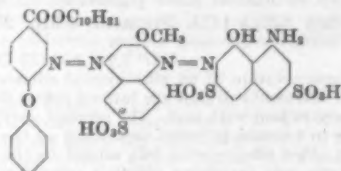
#### Cyan Dyes for use in the Silver-dye-bleach Process of Colour Photography

Gy BP 847,527 (29 Apr 1957)

Dyes of formula—



(A = residue of a diazotized aminoaryl carboxylic alkyl ester (Alk of  $> 4$  C); R<sup>1</sup> = Alk or carboxymethyl; X = OH or NHR<sup>2</sup> (R<sup>2</sup> = H or R<sup>3</sup>CO- (R<sup>3</sup> = unsubst. or Hal-subst. Alk, aralkyl, Ar); n = 1 or 2) e.g.—

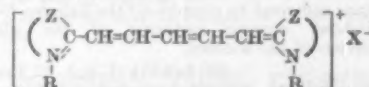


remain unchanged in hue at different levels of bleaching. C.O.C.

#### Dicarbocyanine Dyes

General Aniline BP 849,185 (19 Feb 1959)

Dicarbocyanine dyes of formula—



(Z = atoms to complete a 5- or 6-membered ring, which may be substituted; R = Alk or aralkyl, X = anion) are obtained by reaction of quaternary salts of heterocyclic nitrogenous compounds containing a reactive CH<sub>2</sub> group in the  $\alpha$ - or  $\beta$ -position to the N atom, in presence of an alkaline condensing agent, with the malonaldehyde diacetals of BP 642,830. This is an improvement over previously known methods for these dyes and gives pure compounds. Thus, 2-methylbenzthiazole ethiodide and malonaldehyde diethyl ethyl methyl diacetal in pyridine give the dye where X = I<sup>-</sup>, R = C<sub>6</sub>H<sub>5</sub>, and Z = atoms to form a benzthiazole residue. A.T.P.

#### Alumina-Silica Pigment

Columbia-Southern Chemical Corp.

USP 2,920,974 (15 Feb 1957)

Aluminium silicate-silica compositions particularly effective as rubber pigments and for making paper opaque are obtained by treating an aluminium salt with a mixture of an alkaline-earth metal silicate and silica. The mixture contains  $< 5$  moles of SiO<sub>2</sub> per mole alkaline-earth metal oxide. C.O.C.

#### Coloured Pearlescent Pigments

Juliette Marglen BP 850,449 (U.S.A. 26 Nov 1957)

Formation of a colorant *in situ* on natural or artificial pearlescent flakes or crystals results in a coloured pearlescent product. Thus CdS or phosphomolybdate-dye complexes can be formed on Pearl Essence (C.I. Natural White 1) and the phosphate molybdate complex of FD&C Green No. 1 (C.I. Acid Green 3) on nacreous lead



subcarbonate or bismuth oxychloride. The coloured pearlescent pigments are used in lacquers, paints, etc.

C.O.C.

#### Anhydrite (C.I. Pigment White 25)

National Lead Co. BP 848,617 (U.S.A. 26 Dec 1957)

Gypsum is made into a 20–50% aqueous slurry of particle size < 200 mesh. Anhydrite seed is prepared by rapidly stirring 5–20% of the slurry in 74–80%  $H_2SO_4$  at < 70°C. Gypsum (as  $CaSO_4$ ), 0.0125–1.0 part per part of  $H_2SO_4$  (as 60° Bé acid), is added per minute and the temperature kept at 20–80°C. Finally the remainder of the slurry is added and the mixture heated to 80–100°C to convert all the gypsum to Anhydrite. The product is finely divided pigmentary grade Anhydrite.

C.O.C.

#### Treatment of Acidic Carbon Blacks (C.I. Pigment Black 6 and 7)

Sherwin-Williams Co. USP 2,920,976 (24 Feb 1953)

Charmel impingement blacks and all true acidic Lamp Blacks of pH > 6 are treated with a solution of a salt of a heavy metal and an aliphatic carboxylic acid, e.g. Pb trimethyl acetate, to render them inert to the driers and accelerators normally used in paint manufacture.

C.O.C.

#### Pelleting of Alkaline Earth Silicates

Columbia-Southern Chemical Corp.

BP 848,933 (U.S.A. 5 Dec 1955)

Powdery, soft, amorphous alkaline-earth metal silicates, e.g. Ca silicate, of low bulk density, i.e. 14–16 lb/ft<sup>3</sup>, are converted into denser pellets by merely tumbling them in a container provided that they occupy at least half the volume of it. The products are readily dispersed in water and are very suitable as paper pigments.

C.O.C.

#### Precipitating Silica (C.I. Pigment White 27)

Columbia-Southern Chemical Corp.

USP 2,921,839 (23 Dec 1955)

An aqueous solution of an alkali-metal silicate and an inert water-immiscible liquid are formed into a dispersion and then neutralised with acid. The product varies from a gel granule to a coarse pigment depending on the amount of NaCl or other alkali-metal salt added to the precipitation system, salt favouring pigment precipitation.

C.O.C.

#### Halogen-free Silica Pigments

B. F. Goodrich Co. BP 848,673 (U.S.A. 22 Dec 1955)

Light-coloured to white powdered amorphous silica containing up to 3% of halogen is treated with a liquid or gaseous alcohol free of amine groups at a pressure and temperature sufficient to remove all the halogen from the silica without causing sintering of the silica or decomposition of the alcohol.

BP 848,674 (U.S.A. 22 Dec 1955)

Brown, powdered, disproportionated silicon monoxide ( $SiO_x$ ) ( $Si$ ), ( $x$  and  $y$  = whole numbers) is heated at > 600°C but below fusion point with sufficient gaseous halogen to form a light-coloured to white product. This is then treated with water or preferably steam, in amount and at a temperature sufficient to remove all the halogen without fusing the silicon dioxide.

BP 848,675 (U.S.A. 22 Dec 1955)

A compound containing reactive hydrogen attached to C is used instead of the alcohol. The amount used is such that a residue of the nitrogenous compound is left chemically attached through N to the silica. Examples of soluble compounds are methylamine and ethylamine.

C.O.C.

#### Agglomerating Powdered Pigments

Goodyear Tire & Rubber Co. USP 2,921,931 (9 Oct 1957)

Free flowing powders which are readily incorporated into rubber mixes are obtained by mixing an aqueous slurry of the pigment with a little of a natural or synthetic rubber latex so that the two are blended before the latex coagulates.

C.O.C.

Performance of Titanium Dioxide Pigments in a Sand Grinder (I p. 27)

Powder of Uniform Particle Size (I p. 27)

Relationship of Molecular Structure and Pigments to Coating Performance (V this page)

Aqueous Slurries of Metallic Oxides (V p. 39)

Study of Difficultly Dyeable Fibres. XI—Condensation Dyes of the Diphenylmethane Series I (VIII p. 42)

Colour Couplers (IX p. 45)

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Colours for Plastic Floorings (XIII p. 49)

$R_f$  Values of Some Anthocyanins and Anthocyanin Intermediate Glycosides (XIV p. 50)

Stable Diazo Salts for Chromatographic Spray Reagents (XIV p. 50)

Bibliography on Molecular and Crystal Structure Models (XV p. 52)

## V—PAINTS; ENAMELS; INKS

### Influence of Wet-ground Mica (C.I. 77019) on Acrylic Latex Exterior House Paints

M. Kronstein, R. M. Sonshine, and J. A. Castronoro

Wet Ground Mica Assoc., Tech. Bull., 1 (41) (1960) 5 pp.

Chem. Abs., 59 (25 Aug 1960) 16862

Wet-ground 225-mesh mica in acrylic latex paint gave excellent water-vapour sealing properties, gloss retention and colour retention, weatherometer exposure test and film density in light transmission. Physical and chemical properties, formulation procedures and best performance of wet-ground mica in acrylic latex paints are included.

C.O.C.

### Influence of White Pigments on the Quality and Durability of Oil Paints. I—Drying Oil as Binder

V. J. D. Rascio and W. O. Bruzzoni

Ind. y quim. (Buenos Aires), 29 (1959) 343–348

Chem. Abs., 54 (25 Aug 1960) 16863

White paints of linseed oil to withstand more than 12 months' weathering require ZnO or  $TiO_2$  (C.I. Pigment White 4 and 6) as pigment, of which 30% can be Barytes (C.I. Pigment White 22). White Lead and Lithopone (C.I. Pigment White 1 and 5) are suitable only for indoor use.

C.O.C.

### Relationship of Molecular Structure and Pigments to Coating Performance

H. F. Payne

Corrosion, 16 (6) (1960)

Tech. Topics, 32, 34, 37–38

Chem. Abs., 54 (25 Aug 1960) 16863

Discussion of the main components of coatings with emphasis on their characteristics related to these components. Molecular structure, significance and function of primary and secondary bonds and connection between mol. wt. and performance are considered. Functions of modifiers and how they influence the characteristics of coatings are explained. The mechanism of adherence is briefly reviewed by considering the fundamental mechanism and electrochemical factors involved.

C.O.C.

### Influence of $\gamma$ -Radiation and Neutrons on the Properties of Lacquer Coatings

Z. Jedliński, Z. Hippe and T. Uminski

Przemysł Chem., 39 (1960) 110–112

Chem. Abs., 59 (25 Aug 1960) 16866

Coatings of chlorinated rubber, chlorinated polyvinyl chloride, polyvinyl butyral, copolymer of vinyl chloride and isobutyl ether of polyvinyl alcohol, modified alkyd resins, polymerised linseed oil, and modified phenol-formaldehyde resin were exposed for 26 h to  $\gamma$ -radiation of 10, 800 r at 18–20°C and evaluated by change in properties, e.g. impact-strength, elasticity and adhesion. The radiation contained a neutron beam of mean intensity 5 neutrons/sq.cm/sec. Coatings of polyvinyl butyral, copolymer of vinyl chloride and isobutyl ether of polyvinyl alcohol, alkyd resin, and polymerised linseed oil had better elasticity, and impact strength after irradiation than those aged in air. The coatings containing chlorinated rubber, chlorinated polyvinyl chloride, or modified phenol-formaldehyde resin become rigid and less adhesive.

C.O.C.

#### PATENTS

### Pigment Pastes for use in Coating Compositions

General Aniline

BP 853,218 (U.S.A. 22 Apr 1958)

A dry, powdered pigment is mixed with a compound of formula—



(R = H, Alk or alkenyl of < 5 C), e.g. 2 pyrrolidone. The amount of pyrrolidone compound used should be at



least sufficient to yield a non-breaking paste. The paste, which contains only the two components, requires only stirring and not grinding before being added directly to paints, lacquers, printing inks, etc. C.O.C.

#### Dispersing Powders in Non-aqueous Liquids

A. L. Veidier BP 850,189 (France 2 Aug 1957)

Inorganic salts and oxides are produced as dry powders in a confined atmosphere, either by dry grinding or by chemical reaction in a gas. They are then gradually dispersed in an amount of the liquid (whose b.p. must be  $> 200^{\circ}\text{C}$ ) several times larger than that corresponding to the composition of the final product. Dispersion is effected in an atmosphere in sealed communication with the confined atmosphere in which the particles were formed. The surplus liquid is then removed by mechanical means until the dispersion reaches the desired composition. This method ensures perfect dispersion of pigments, fillers, etc. C.O.C.

#### Dispersing Solids in Liquids

Torrance & Sons BP 850,687 (13 July 1956)

When preparing paints containing China Clay,  $\text{BaSO}_4$  or  $\text{CaCO}_3$  (C.I. Pigment White 19, 21, 22 and 18) a dispersion of finer particle size is obtained by ball milling using stearate beads as the dispersing elements. The diameter of the beads is preferably 2.5–4.5 mm.

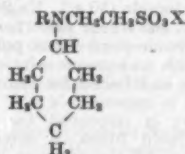
BP 850,688 (13 July 1956)

The solids and the liquid are intermittently added while the mill is running. The dispersion is also intermittently withdrawn in like quantities while the mill is running. The operations of addition and withdrawal alternate with a period between each, the mill being run continuously. This gives a dispersion of high quality. C.O.C.

#### Dispersing Solid Pigments in Organic Liquids or Solids

General Aniline USP 2,919,993 (31 Dec 1956)

Compounds of formula—



(R = acyl of 12–18 C; X = alkali metal or ammonium) are used as the dispersing agent, e.g. in the making of paint or crayons. Thus molten paraffin wax (9 parts) had added to it a 20% active aqueous slurry of Na N-palmitoyl-N-cyclohexylsulfate (0.1) and then Barytes (C.I. Pigment White 22). It was then well stirred. A similar sample was prepared without use of the dispersing agent. Eight minutes after stirring ceased, although in each case much of the Barytes had settled out, the supernatant liquid of the sample containing the dispersing agent was cloudy, whereas that of the other sample was clear. C.O.C.

#### Anti-sedimentation Agents for Pigment Suspensions

Dehydag Deutsche Hydrierwerke

BP 850,878 (Germany 29 June 1957)

The settling and cementing of pigment suspensions, e.g. paints and lacquers, is retarded by addition of 0.05–0.2% of neutral salts or salt mixtures of deriv. of sulphosuccinic acid  $\text{R-X-CO-CH}_2\text{-CH}_2\text{-COOY}^1$  (either  $\alpha$ -C or  $\beta$ -C is subst. by  $\text{-SO}_3\text{Y}^2$ ; R = lipophilic hydrocarbon residue of 8–18 C, which may be subst. by a hetero atom or hetero atom group; X = imido, possibly organically substituted;  $\text{Y}^1$  and  $\text{Y}^2$  = same or different inorganic or organic cation). Salt mixtures containing one or more metallic, preferably divalent bases, e.g. Na, K, Mg, Ca, Zn, and also  $\text{NH}_4$ , or one or more, preferably tertiary, bases, e.g. pyridine, triethanolamine, are preferred. They are obtained by condensing amines of 8–18 C, or mixtures thereof, with maleic anhydride at  $< 50^{\circ}\text{C}$ . The resulting alcoholic soln. of maleic acid monoalkyl amide is mixed with an aq. soln. containing an amount of bisulphite equivalent to the amide and is then neutralised. The products are added directly to pigment suspensions before or after diluting, either as solid or as a conc. soln. or paste, or by dry grinding with the pigment. A.T.P.

#### Surface Coatings of Unsaturated Polyester-Ethynically Unsaturated Monomer Copolymers

Jenson & Nicholson Group BP 852,036 (12 Mar 1958)

Coatings prepared from di- or poly-hydric alcohols and di- or poly-carboxylic acids, at least one of the components being unsaturated, tend to become soft and tacky. This is overcome by treatment with a solution of wax or fat in a volatile solvent within  $> 2$  h after the coating has been applied. This gives far better results than adding the wax to the coating composition, as all the wax coating is removed when the hardened coating is rubbed down. C.O.C.

#### Aqueous Slurries of Metallic Oxides

United States Atomic Energy Commission

BP 851,983 (U.S.A. 10 Mar 1958)

The rate of settling of metallic oxides in aqueous slurries is much reduced if the oxide particles are coated with an organosilicon compound and then fired at a temperature which decomposes the organosilicon compound but does not cause the oxide particles to sinter. C.O.C.

#### Enamels

Dr. Richmann & Rappe

BP 849,481 (Germany 30 July 1956)

Enamels containing  $> 10\%$  Al, 0–12% Si and  $< 3\%$  P have a similar m.p. and as high resistance to acid as normal enamels of low Al and high Si content. C.O.C.

#### White Coating Composition suitable for Facing Concrete

Klöcker-Hütte Bremen

BP 849,175 (Germany 6 Nov 1957)

A mixture of a white cement, e.g. Portland cement, (3 parts by wt.) and white additives (10) is used;  $< 3\%$  of the white additives is  $\text{TiO}_2$  (C.I. Pigment White 6). C.O.C.

Mill for Ink, Paint, and the like (I p. 27)

Coating Compositions comprising a Thermoplastic Acrylic Copolymer and a Diglycidyl Ether (III p. 29)

Treatment of Acidic Carbon Blacks (C.I. Pigment Black 6 and 7) (IV p. 38)

Epoxy Ester Coatings for the Metal Litho Industry (X p. 46)

Penetration of Varnishes and Inks into Paper under Pressure (XI p. 47)

Effect of Driers on Drying Times of Clear and Pigmented Alkyd Resins (XIII p. 48)

Rapid Analysis for Quality Control of Paint Driers (XIV p. 50)

## VI—FIBRES; YARNS; FABRICS

#### Degradation of Cellulose by Hypochlorite Treatment

O. Samuelson and L. A. Wikström

Svensk Papperstidning, 63 (15 Sept 1960) 543–549

Purified raw cotton was treated with sodium hypochlorite soln. containing ca. 1.0 g/l. available chlorine at pH 10.0 and 11.6, and at  $45^{\circ}\text{C}$ , followed by mercerising. The carboxyl and carbonyl contents and the DP (osmotic pressure) were then measured, together with the content of soluble acids. At pH 10.0 the carbonyl content rose from 0.06 to ca. 0.31 m.equiv./100 g in 10 h and then remained constant, whereas the carboxyl content rose progressively from 0.23 to 4.27 m.equiv./100 g over 48 h. In the same time DP fell to 260–270. Results at pH 11.6 were similar except that the carbonyl content passed through a max. of 0.44 m.equiv./100 g after 24 h. Most of the available chlorine consumed resulted in the formation of soluble material. A.J.

#### Iodine Sorption on Cellulose Fibres

H. Tanzawa

J. Soc. Text. Cellulose Ind. Japan,

16 (May 1960) 373–380

Equilibrium sorption isotherms of iodine on cellulose fibres determined in aqueous solution in presence of KI at  $25^{\circ}\text{C}$  fit the Langmuir equation. Thus the parameters of this equation can be used to measure the degree of crystallinity and accessibility. The method is, however, not convenient for estimating the degree of crystallinity but is useful for detecting variation in the fine structure of the newly regenerated cellulose gel when it is dried or stretched. C.O.C.

### Mechanical Properties of Wool Keratin and its Molecular Configuration

M. Feughelman and A. R. Haly

*Kolloid Z.*, 168 (2) 107-115 (in English)  
*Chem. Abs.*, 54 (25 Aug 1960) 16842

The relationship between the amount of supercontraction at the end of the first and final stages of a wool fibre in cone. LiBr and the mechanical properties of the fibre at these stages in the LiBr solution is explained in terms of a simple structural model. This requires two types of zones of different thermal stability along the microfibrils in the keratin. One zone, comprising about 36% of the microfibrils, unfolds unimpeded by bulky side chains, and the remaining 64% may be unfolded by overcoming steric hindrance and causing irreversible breakdown of bonds. The model can be used to explain the load-extension behaviour of single wool fibres. It may also be applied to other mechanical behaviour, e.g. creep in the yield and post-yield regions, and to the second-order transition temperature observed in wool stretched in water.

C.O.C.

### Supercontracting and Setting Behaviour of Modified Wool Fibres

A. R. Haly and M. Feughelman

*Text. Research J.*, 30 (May 1960) 365-372

Ultraviolet irradiation of wool increases the rates of supercontraction in unbuffered aq. LiBr and of setting in boiling water. A second slower effect is shown by progressive reduction in degree of contraction at equilibrium. Treatment with thioglycolic or peracetic acid also increases these rates, whereas iodination or treatment with Van Slyke reagent reduces them. There is little or no loss of tyrosine and little loss of cystine during irradiation to obtain maximum effect in the rate of supercontraction. Rate of supercontraction, rate of setting and second-order transition temperatures are closely related, as are equilibrium contraction, maximum set and remanent stress. 34 References.

C.O.C.

### Reactivity of the Morphological Components of Heat-treated Wool

C. Daveloose, G. Mazingue, and M. van Overbèke

*Bull. Inst. Text. France.* (88) (May-June 1960) 61-75

Swelling in ammoniacal KOH, the Allwörden reaction, and solubilisation in cupriethylenediamine and in sodium sulphide, all indicate that the effect of heat on wool is to decrease the reactivity of the ortho-cortex and to increase that of the para-cortex—i.e. the cortical asymmetry of wool is decreased by heating. The reasons for this behaviour are discussed.

J.C.F.

### Chemical Modification of Wool by Trichloroacryloyl-diazomethane and 1,4-Dichlorobut-2-yne

A. Kling *Melliand Textilber.*, 41 (June 1960) 729-732

Wool treated with 1,4-dichlorobut-2-yne is somewhat more resistant to bacterial attack than untreated wool. Treatment with  $K_2O$ ,  $r_2O_2$  is, however, much more effective.

W.M.

### Studies on the Stiffness of Silk Fibres

VII—Softening of Raw Silk by Urea

H. Takagi

*J. Soc. Text. Cellulose Ind. Japan*,

16 (May 1960) 366-369

When raw silk is treated with urea, the greater the strength of the urea solution the greater the weight-increase ratio (the relation is almost linear) and the greater the softening effect. Time of treatment has no influence on the softening effect. The higher the temperature of treatment and/or the lower the degree of dehydration after treatment, the greater are the weight-increase ratio and the softening effect. Hardly any difference is observed in the softening effect with raw silks of differing deniers. Treatment with urea causes no extraordinary shrinkage. Standing baths of urea can be used without any appreciable effect on the softness produced. Interaction between urea and sericin is almost the same as that between water and sericin.

### VIII—Effects of Urea on Sericin

*Ibid.*, 369-372

Interaction between urea or cationic surfactants and silk is not only by chemical adsorption on the surface. The bonds between sericin and cationic surfactants are fairly strong, whereas those between sericin and urea are

comparatively weak. Softening of raw silk with urea must be done in an atmosphere of fairly high humidity.

C.O.C.

### Man-made Fibres. Some Recent Developments—I

A. J. Hall *Fibres & Plastics*, 21 (Nov 1960) 330-333, 341  
A review in which are discussed, *inter alia*: modification of cellulose acetate, improvements in dyeing properties of cellulose triacetate materials, and improvement in dye-uptake of acrylic fibres by inclusion of polyvinyl pyrrolidone, which is treated at considerable length.

J.W.D.

### Manufacture and Dyeing of Polyamide Fibres—I

R. S. Asquith and G. E. Styan

*Fibres & Plastics*, 21 (Nov 1960) 323-326

Factors leading to the present method by which nylon 6.6 is produced are outlined. Research and development during recent years are discussed under two headings: improved methods for production of nylon 6.6, modifications of nylon 6.6 to change its specific properties. Under the latter heading reference is made to the improvement of elasticity by the introduction of bulky side-chains to reduce hydrogen bonding, the increasing of tensile strength by cross-linking, the improvement of electrical conductance (decrease in susceptibility to accumulate static charge) by graft co-polymerisation, and the modification of mechanical and insulating properties by producing yarns with hollow and irregular cross-sections.

J.W.D.

### Tygaflor PTFE-coated Glass Fabrics and Yarns

*Rubber & Plastics Age*, 41 (Sept 1960) 1047

Account of properties and some applications of a range of glass fabrics and yarns (Fothergill and Harvey) coated with polytetrafluoroethylene.

J.W.D.

### Carbamoylethylation of Polyvinyl Alcohol Fibres with Acrylamide

H. Ito

*J. Soc. Text. Cellulose Ind. Japan*,

16 (May 1960) 381-387

When polyvinyl alcohol fibres are treated in an aqueous solution of acrylamide (30 g/l.,  $Na_2SO_4$  (250),  $Na_2PO_4$  (20) for 4 h at 60°C, the liquor ratio being such that there is 1 mol. of acrylamide per 0.6 mol. polyvinyl alcohol, or are impregnated with an aqueous solution of acrylamide (350) and NaOH (70) and baked for 5 min at 120°C, the degree of substitution is approx. 2-4 mol. % in both cases. As elastic recovery is improved by formalising partially carbamoylethylated fibres, a certain degree of intermolecular cross-linking would seem to occur. In addition, the Mannich reaction is observed when partially carbamoylated fibres are treated with amines. The after-treated fibres have improved affinity for some dyes, particularly good results being obtained when the fibres are aftertreated with dimethylamine and then formalised.

C.O.C.

### Glass—The Oldest and the Newest Fibre

H. V. Blake

*Fibres & Plastics*, 21 (Nov 1960) 327-328, 341

An account of the manufacture, properties, and uses of glass fibres. The last of these is treated under four headings: electrical insulation, reinforced plastics, coated fabrics and paper reinforcement, and decorative fabrics.

J.W.D.

### PATENTS

#### Modified Manilla Hemp

Fabric Research Laboratories BP 853,629 (13 Feb 1957)

Manilla hemp is treated in 10-15% NaOH for 5-30 min at 0-30°C, neutralised and then treated in 0.25-1.0% aqueous methacrylate chromic chloride at pH 3-5 at room temperature for 30 min-20 h, dried and baked for 20 min at 300-350°F. This imparts permanent and marked increase of elongation, e.g. several hundred per cent, when either dry or wet and has little effect on tensile strength.

C.O.C.

#### Rendering Synthetic Polymer Yarns Non-slipping

Inventa AG für Forschung und Patentverwertung

BP 853,325 (Switzerland 20 June 1957)

The yarns are treated with polyvinyl alcohol and 3-hydroxy-n-butylaldehyde in presence of an acid catalyst and preferably in presence of a swelling agent for both the fibres and the resin being produced. The coating so produced in the fibres is very resistant to mechanical influences.

C.O.C.

**Non-fibrillating Acrylic Fibres**

Chemstrand Corp. BP 848,696 (U.S.A. 19 Dec 1955)

Fibres of polyacrylonitrile or blends of it with polymers of mono-olefinic monomers are treated at high temperature and pressure in presence of saturated or wet steam. This removes their tendency to fibrillate. C.O.C.

**Acrylonitrile Copolymers having Enhanced Dyeability**

Dow Chemical Co. BP 850,435 (U.S.A. 24 Jan 1958)

The affinity of acrylonitrile copolymers for a wide variety of dyes is increased by incorporating an *N*-vinyl-3-morpholinone polymer. W.G.C.

**Acrylic Fibres of Enhanced Dye Affinity**

Eastman Kodak Co. USP 2,921,042 (7 Apr 1954)

An aqueous dispersion of bentonite is treated with an aqueous solution of an amine salt of  $> 9$  C. This renders the bentonite hydrophobic and readily dispersible in solvents. A dispersion of the treated bentonite, e.g. dimethylformamide, is added to an acrylic polymer spinning solution so as to incorporate 8–15% bentonite in the resulting fibres. This gives them greatly enhanced affinity for disperse, acid, vat, and direct dyes. C.O.C.

**Synthetic Nitrogenous Polymers for making Filaments and Films**

DuPont BP 849,154 (U.S.A. 29 Dec 1955)

Polymers having recurring  $-N-N-$  groups ( $X^1$  and  $X^2$ )

$X^1 = H$  or hydrocarbon) linking organic radical of mol. wt. 700–8000 and of formula  $-OC-Y-CO-$  ( $Y =$  number of recurring units of which  $-OC-Y-CO-$  radicals form at  $\leq 60\%$  by wt.), are elastomers of good mechanical properties and stability to ultraviolet radiation. They are very suitable for use in producing white elastic fibres. C.O.C.

**Improving Dyeing Properties of Polymers containing Nitrile Groups**

Gevaert Photo Produkten

Modification of BP 786,960, even better dyeing properties being obtained if, after treatment with hydroxylamine, the product is treated first with an acid and then with a base. C.O.C.

**Improving the Water Absorption of Nylon**

FBY BP 847,194 (Germany 28 Sept 1957)

Modification of BP 788,688 (J.S.D.C., 74 (1958) 212) by using instead of an *N*-methoxymethyl polyamide another *N*-alkoxyalkyl polyamide, *N*-hydroxyalkyl polyamide, or *N*-alkthioalkyl polyamide, e.g. *N*-ethoxymethyl polyamide, *N*-hydroxymethyl polyamide and *N*-(alkylthiomethyl) polyamides. C.O.C.

**Polycarbonate Fibres**

FBY BP 844,488 (Germany 17 Jan 1957)

Yarns of high strength are produced by stretching a crystallisable polycarbonate 20–30 deg. C above the conversion temperature. W.G.C.

**Polymeric Polymethylene Terephthalates incorporating a Fluorescent Brightening Agent**

ICI BP 851,792 (14 Mar 1958)

A fluorescent brightening agent, preferably containing a group capable of reacting with OH or COOH groups, e.g. 2,8-di(2,6-dimethylphenyl)-anthra-1',9'(N), 5',10'(N)-dipyridazone, is incorporated into the polymer at any stage of its manufacture, e.g. into the polymerisation mixture or the molten polymer. This allows a polymer of satisfactory colour to be produced commercially from glycol and terephthalic acid which has not been purified to the degree that would be necessary if the fluorescent brightening agent were not used. C.O.C.

**Glass Fibres Partly Coated with Metal**

Owens-Corning Fiberglass Corp.

BP 847,183 (U.S.A. 11 Sept 1957)

Glass filament having a continuous metallic coating on only part of its periphery, preferably not more than half, has higher lustre than a filament having a complete coating. Yarns made from such filaments have greater brilliance and higher lustre than other metallic yarns. C.O.C.

**Polyester Fibres Stabilised to Ultraviolet Radiation**

(X p. 47)

Improving the Affinity for Dyes of Polymers or Copolymers of Ethylene and Propylene with Simultaneous Imparting of Lyophilic and Antistatic Properties (XIII p. 49)

**VII—DESIZING; SCOURING; CARBONISING; BLEACHING**

PATENTS

**Desizing Cellulose Fabrics**

Sociétés d'Études Chimiques pour l'Industrie et l'Agriculture BP 843,559 (France 10 July 1956)

A satisfactory and inexpensive method of desizing is to treat fabrics with alkali-metal bromites and/or hypobromites at pH  $> 9$ . This results in total removal of all amylaceous substances, even in very tightly woven fabrics, in a short time at ordinary temperatures. Thus cotton poplin containing 12.2% size is given two runs through an aqueous solution of Na bromite (5 g active Br/l.) and a wetting agent, the pH of the liquor at the start being 9.8. The cloth is given two runs through the liquor at 30 m/min, being squeezed off after each run to a 50–55% liquor content. It is then worked in 0.5% aq. NaOH at 90°C and secured. C.O.C.

**De-oiling and Desizing Regenerated Cellulose and Cellulose Ester Fibres**

ICI BP 844,943 (13 Feb 1957)

The goods are first treated with trichloro- or perchloroethylene. Entrained solvent is then flashed off by immersing the goods in hot water. Finally they are treated with an aqueous solution of a peroxygen compound and steamed. C.O.C.

**Bleaching Cotton**

Montecatini Società Generale per l'Industria Mineraria e Chimica BP 844,699 (17 Dec 1956)

The goods are impregnated with an aqueous solution of an alkali-metal hydroxide, alkali-metal silicate,  $H_2O_2$ , and a wetting agent. They are then macerated and steamed. This yields goods of 90–92% whiteness, a complete white being obtained by subsequent conventional bleaching. Thus cotton cloth was impregnated with its own weight of a soln. of NaOH (4 g/l.), Na silicate (84), 130 vol.  $H_2O_2$  (15 ml/l.) and wetting agent (6 g/l.). After standing for 3 h it was steamed at 100°C for 15 min. This yielded cloth ready for dyeing or for conversion to a full white by normal peroxide bleaching. C.O.C.

**Bleaching with Permonosulphuric Acid**

Deutsche Gold- und Silber-Scheideanstalt

BP 845,063 (Germany 6 Sept 1957)

When bleaching with permonosulphuric acid, addition of 0.5–5.0 parts of oxalic acid per 10 parts of permonosulphuric acid reduces the loss in strength to 25–10% of what it would be in absence of the oxalic acid. Thus cotton poplin was treated at 90°C for 2 h and 1:40 liquor ratio in a bath containing  $KHSO_5$  (5 g/l.) and oxalic acid (1). The fabric lost 7.2% in strength, whereas a control similarly treated in absence of oxalic acid showed a 46% strength loss. C.O.C.

**Bleaching Continuous-spun Viscose Rayon**

T.M.M. (Research) BP 849,858 (3 June 1958)

On a continuous spinning machine, viscose rayon is bleached by successive treatments with an aqueous acid solution, an aqueous solution of an alkali-metal permanganate, and an aqueous solution of hydrogen peroxide. W.G.C.

**Dry-cleaning**

Boehme Fettechemie BP 847,017 (Germany 27 Apr 1957)

Greatly improved cleaning is obtained if the solvent contains, in addition to the usual surfactants, water and 0.1–10 g/l. of amines of 8–32 C, e.g.  $\beta$ -aminoethyldecyl ether or naphthylamine. C.O.C.

**Dry-cleaning**

Printar Industries BP 848,483 (18 Oct 1957)

Dispersing formalin in the solvent with the aid of a dispersing agent results in disinfection simultaneously with cleaning. Such dispersions are especially useful for treating blankets and other wool goods. A mothproofing agent



and/or a heavy petroleum product (to stop fluff formation) may also be present. C.O.C.

Degradation of Cellulose by Hypochlorite Treatment (VI p. 39)

Studies in Adsorption. XI—System of Classification of Solution Adsorption Isotherms, and its Use in Diagnosis of Adsorption Mechanisms and in Measurement of Specific Surface Areas of Solids (VIII below)

Preventing Reddening of Unbleached or Semi-bleached Sulphite Pulp (XI p. 48)

Energy of Aqueous Wetting of Bulk Surfaces (XIV p. 52)

## VIII—DYEING

Studies in Adsorption. XI—System of Classification of Solution Adsorption Isotherms, and its Use in Diagnosis of Adsorption Mechanisms and in Measurement of Specific Surface Areas of Solids C. H. Giles, T. H. MacEwan, S. N. Nakhwa, and D. Smith J.C.S. (Oct 1960) 3973-3993

Isotherms for adsorption of org. solutes are divided into four main classes, according to the slope of the initial portion of the curve, and thereafter into sub-groups. The main classes are: S Curves, indicative of vertical orientation of adsorbed mol. at the surface; L Curves, the normal or "Langmuir" isotherms, of mol. adsorbed flat on surface, or, sometimes, of vertically oriented adsorbed ions with particular strong intermol. attraction; H Curves ("high affinity") (commencing at a positive value on the "concentration in solid" axis), often given by solutes adsorbed as ionic micelles, and by high-affinity ions exchanging with low-affinity ions; C Curves ("constant partition"), which are linear and given by solutes which penetrate into the solid more readily than does the solvent. The sub-groups of these classes are arranged according to the shape of the parts of the curves farther from the origin, and the significance of plateaux and changes of slope are described. It is suggested that *p*-nitrophenol may be one of the best cpd. for reliable measurements of specific surface areas. 156 Systems are classified. H.H.H.

Thermodynamics of Irreversible Processes in Dyeing I—The Discontinuous System

H. Vedaïra J. Soc. Text. Cellulose Ind. Japan, 16 (May 1960) 403-405

Entropy production in dyeing has been calculated and then the phenomenological equations obtained, the work being based on the assumption that the dyeing system consists of two sub-systems—the fibre phase and the dyebath. The diffusion coefficient of dye within the fibre depends on the zeta-potential of the fibre and increases with increase in concentration of the foreign electrolyte. In the steady state, the rate of adsorption of dye by fibre depends on the difference of the chemical potentials of the inactive component. C.O.C.

Dyeing Behaviour of Disperse Dyes on Hydrophobic Fibres

V. S. Salvin

Amer. Dyestuff Rep., 49 (22 Aug 1960) 600-605, 608

Work which shows the solution theory for the dyeing of cellulose triacetate with disperse dyes is valid. Diffusion rate within the fibre is the rate-controlling factor. The kinetics of dyeing of hydrophobic fibres, including triacetate and polyethylene terephthalate, have been related to changes in accessibility of the fibre to the dye and to the mobility of the dye. The attractive forces holding dye to fibres are discussed as they affect the diffusion rate. Carriers make the fibre more accessible because the adsorbed carrier loosens the fibre bonds. Thus carrier activity replaces high temperature in effecting diffusion. The specificity of dyes for polyester fibres as against cellulose triacetate is shown. The hydrophobic nature of the dyes having the better dyeing properties on polyester fibres suggests further work on the relation of diffusion rate to saturation value and on the contribution of Van der Waals forces to affinity. C.O.C.

Dyeing Polyester Fibres using Carriers

P. Rochas Chemiefasern, 10 (Oct 1960) 666-670

Polyester fibres of highly orientated and close-packed structure are difficult to penetrate by dye molecules and, since few active groups are accessible, their uptake of dye

is very limited. When dye mol. are of the same order of size or smaller than the inter-micellar spaces and pores, the diffusion rate is greatly increased. With dyes of small mol. size there is some loss of fastness properties, and better results are obtained with dyes of higher mol. wt. applied by special techniques. Heat increases mol. motion and this is most marked in cryst. regions of the fibre; this opens up the fibre structure and greatly assists diffusion. Dyeings of equal depth are obtained in 11 s at 200°C, 5 min at 125°C, and 5 days at 60°C. Disperse dyes of high mol. wt. can thus be applied at high temp. Dye uptake can be greatly accelerated by use of carriers, so that medium and dark shades can be attained in commercially practicable times. Carriers associate strongly with the polymer chains, not with the dye, probably forming a monomolecular layer; they act as softeners, opening the fibre structure and making it more accessible to dye. This supplements the action of heat. The resultant rearrangement of the polymer chains can produce a shrinkage of as much as 10%, it is confined almost entirely to the amorphous regions of the fibre, and it does not lead to an increase in susceptibility to swelling in aq. media. Optimum action of the carrier requires only low concn. within the fibre—ca. 70 mg/g of fibre. If more is present, three types of behaviour may obtain: (a) fixed dye increases to a max. value with increasing carrier content and then decreases rapidly, e.g. with diphenyl as carriers; (b) fixed dye rises to max. value at ca. 70 mg/g of fibre (same value as in (a) above and (c) following), remains constant up to carrier content of ca. 200 mg/g of fibre, and then decreases rapidly, e.g. with *o*-phenylphenol as carrier; or (c) fixed dye rises to max. value and reaches saturation, e.g. with methyl salicylate as carrier. With a carrier, uptake of dye is greater the slower the uptake when carrier is absent. Carriers are more effective with disperse dyes of azo type than with those of anthraquinonoid type; this may be because the mol. configuration of azo dyes fits that of the polyester chains better. The saturation quantity of carrier taken up by polyester fibres, which is independent of the concn. of the bath, corresponds to the complete coverage of the chain surfaces in the amorphous regions.

A two-bath method is recommended in which the carrier is applied from the first bath (concn. of which should be enough to ensure uptake of the optimum 70 mg carrier per g fibre), excess carrier—which might affect adversely the subsequent dyeing—is removed by thorough washing with hot water containing, if necessary, a neutral surfactant, and dyeing proper is effected in the second bath. The carrier represented by the optimum uptake is very tightly bound to the fibre and resists removal by either hot water or solvents such as acetone, but it is completely removed by alkaline treatment before the intermediate wash. Bath concn. of 12 carriers necessary to ensure optimum uptake are listed. J.W.D.

Constitution and Effectiveness of Organic Substances as Carriers in the Dyeing of Polyester Fibres with Disperse Dyes—(I)

P. Senner and Th. Böhm

Textil Praxis, 15 (Sept 1960) 940-944

After referring to the high-temperature and Thermosol methods of dyeing polyester fibres, the author discusses briefly advantages and drawbacks of carrier dyeing. In the experimental part, the behaviour of 1,4-dimethylaminoanthraquinone (Celliton Fast Blue B (BASF)) in carrier dyeing is described in some detail. In order to facilitate and simplify estimation of the dye on the substrate, specially prepared polyester film was used in all dyeing experiments. Dyed samples were measured directly in a Lange colorimeter, and they were also used in subsequent light exposure tests. The effect on the dye uptake of dyeing time, dye concentration, carrier concentration, and temperature is depicted by means of dyeing curves. L.A.T.

Study of Difficultly Dyeable Fibres. XI—Condensation Dyes of the Diphenylmethane Series I

K. Yamada J. Soc. Text. Cellulose Ind. Japan,

16 (May 1960) 412-417

Use of 4,4'-bisdimethylaminodiphenylmethane (I) instead of 4,4'-bisdimethylaminobenzhydrol with chloranil, *p*-nitrosodimethylaniline and aromatic phenols in the condensation-combination dyeing of Vinyon is simpler

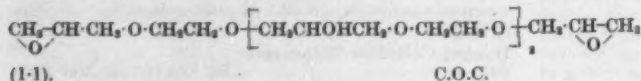




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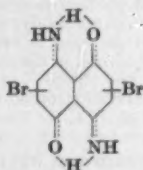
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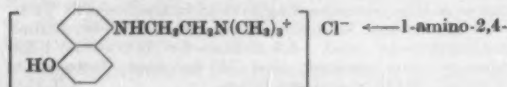
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A reducible silver salt image is developed with an aromatic primary amino developer or with an amino aromatic developer which splits in the alkaline developing solution to yield the corresponding amine, in presence of a carboxylic acylacetanilide colour coupler the anilide portion of which is substituted in 2-position by alkoxy and in 5-position by an alkyl-, aryl- or substituted arylsulphonamide group, e.g. 3-acetamino-4-methoxybenzene-n-butylsulphonamide. These couplers are more reactive than the hitherto known sulphonamide-substituted acylacetanilides. C.O.C.

#### Multi-colour Printing Machine (I p. 27)

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#### Multi-colour Printing on Stretchable Fabrics (I p. 27)

#### Cyan Dyes for use in the Silver-dye-bleach Process of Colour Photography (IV p. 37)

#### Pad Dyeing or Printing of Textiles with Pigments (VIII p. 43)

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#### Dyeing and Printing Polymers and Copolymers of Acrylonitrile and Asymmetric Dicyanoethylene (VIII p. 44)

#### Carbon Paper producing Copies for Thermal Copying (XI p. 48)

## X—SIZING AND FINISHING

### Stenter Drying and Heat Treatment of Fabric

K. S. Laurie J. Textile Inst., 51 (Sept 1960) p 562-r 565

The principles of cloth guiding and fabric handling in relation to stentering are briefly reviewed. In stenter drying it is essential that air application and drying should be even across the width of the cloth, and the air streams above and below the cloth must be balanced (these precautions also obviate the migration of dyes or impregnated finishes); fabric temp. must not be high enough to cause deterioration. High rates of drying require—(a) max. air temp. consistent with protection of the fabric, (b) lowest reasonable air humidity, and (c) highest possible air-jet speeds at the surface of the fabric. The heat treatment of fabrics by hot-air jets is governed by the same principles as drying, except that air-jet speed is less critically important and temp. control is much more important; the machine should also be accessible for cleaning. The modern design of stenter housings is briefly discussed. J.W.D.

### Hot Air Drying—Effects of Temperature and Humidity

P. Wadsworth

J. Textile Inst., 51 (Sept 1960) p 552-r 562

It is shown that to obtain a high rate of evaporation a high air temp. and a low air humidity or wet-bulb temp. are required. During most of the drying time the cloth is at the wet-bulb temp., but as evaporation proceeds to the limit (e.g. if the cloth remains stationary for a time in the dryer) it reaches the air temp. To avoid cloth damage, air temp. is thus practically limited to 300–330°F. The wet-bulb temp. or the humidity is limited by the thermal efficiency of the process. Most of the heat required by a dryer is for the evaporation of the water in the cloth and this varies only slightly with conditions of evaporation. It is shown that a superheated vapour dryer is the most efficient thermally, but not in relation to rate of evaporation or productivity. The efficiency of many old stenters is greatly reduced by their housing in enclosures that are too large, badly insulated, and far from air-tight. Data are given for 12 stenters, showing the superiority of modern machines in relation to specific steam consumption (lb of steam per lb of water evaporated) and rate of evaporation (lb per sq. ft per h). The re-housing of an old machine on modern lines, reducing the amount of fresh air introduced during operation, more than halved the steam requirement whilst slightly increasing the rate of evaporation. The lowest efficiency on both counts was exhibited by a machine having no air recirculation. J.W.D.

### Improving Cotton Cord by Ozonisation

Kh. V. Usmanov, V. A. Kargin, B. I. Alkhodzhaev, and N. Sh. Inoyatov

Vysokomolekulyarnye Soedineniya, 2 (1960) 88–91

Chem. Abs., 54 (25 Aug 1960) 16841

Medium-staple cotton cord was ozonised for 10 h and then washed with water at 25°C. Its strength was increased stepwise from 7.37 to 7.92 and to 7.95 kg/mm<sup>2</sup>. The fats and waxes were decomposed and completely washed off. The sorptive properties increase on ozonisation and decrease on washing and extraction because the low-molecular substances formed by ozonisation are more hydrophilic than cellulose. C.O.C.

### Micro-organism Growing on Cloth. VIII—Treatment of Cloth containing Cellulose Acetate with 8-Hydroxyquinoline Copper Acetate

K. Ichishima

Kasei-gaku Zasshi, 11 (1960) 40–43

Chem. Abs., 54 (25 Aug 1960) 16840

The adverse effect on cellulose acetate of treatment with 8-hydroxyquinoline Cu acetate is much reduced if the bath is kept at <60°C (at which  $\beta$ -transformation of the fibre occurs). C.O.C.

### Replacing THPC in the APO-THPC Flame-resistant Finish

T. D. Miles, F. A. Hoffman, and A. Merola

Amer. Dyestuff Rep., 49 (22 Aug 1960) 596–599

Compounds other than tetrakis(hydroxymethyl)phosphonium chloride can be combined with tri-1-aziridinyl phosphine oxide ( $\text{CH}_2\text{CH}_2\text{N}$ )<sub>3</sub>PO (APO) as a durable flame-retardant for cotton. They must be able to catalyse the polymerisation of APO. An infrared

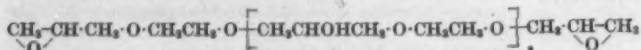


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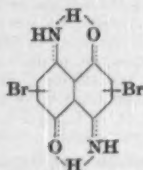
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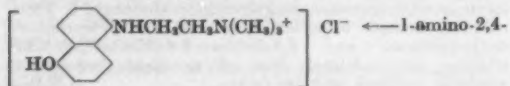


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#### Colour Photography

Gevaert Photo-Producten BP 848,558 (15 Mar 1957)

A reducible silver salt image is developed with an aromatic primary amino developer or with an amino aromatic developer which splits in the alkaline developing solution to yield the corresponding amine, in presence of a carboxylic acylacetanilide colour coupler the anilide portion of which is substituted in 2-position by alkoxy and in 5-position by an alkyl-, aryl- or substituted arylsulphonamide group, e.g. 3-acetamino-4-methoxybenzene-*n*-butylsulphonamide. These couplers are more reactive than the hitherto known sulphonamide-substituted acylacetanilides. C.O.C.

Multi-colour Printing Machine (I p. 27)

Printing Machine (I p. 27)

Multi-colour Printing on Stretchable Fabrics (I p. 27)

Cyan Dyes for use in the Silver-dye-bleach Process of Colour Photography (IV p. 37)

Pad Dyeing or Printing of Textiles with Pigments (VIII p. 43)

Dyeing and Printing Acrylic Fibres (VIII p. 44)

Dyeing and Printing Polymers and Copolymers of Acrylonitrile and Asymmetric Dicyanoethylene (VIII p. 44)

Carbon Paper producing Copies for Thermal Copying (XI p. 48)

## X—SIZING AND FINISHING

### Stenter Drying and Heat Treatment of Fabric

K. S. Laurie J. Textile Inst., 51 (Sept 1960) p 562–p 565

The principles of cloth guiding and fabric handling in relation to stentering are briefly reviewed. In stenter drying it is essential that air application and drying should be even across the width of the cloth, and the air streams above and below the cloth must be balanced (these precautions also obviate the migration of dyes or impregnated finishes); fabric temp. must not be high enough to cause deterioration. High rates of drying require—(a) max. air temp. consistent with protection of the fabric, (b) lowest reasonable air humidity, and (c) highest possible air-jet speeds at the surface of the fabric. The heat treatment of fabrics by hot-air jets is governed by the same principles as drying, except that air-jet speed is less critically important and temp. control is much more important; the machine should also be accessible for cleaning. The modern design of stenter housings is briefly discussed. J.W.D.

### Hot Air Drying—Effects of Temperature and Humidity

P. Wadsworth

J. Textile Inst., 51 (Sept 1960) p 552–p 562

It is shown that to obtain a high rate of evaporation a high air temp. and a low air humidity or wet-bulb temp. are required. During most of the drying time the cloth is at the wet-bulb temp., but as evaporation proceeds to the limit (e.g. if the cloth remains stationary for a time in the dryer) it reaches the air temp. To avoid cloth damage, air temp. is thus practically limited to 300–330°F. The wet-bulb temp. or the humidity is limited by the thermal efficiency of the process. Most of the heat required by a dryer is for the evaporation of the water in the cloth and this varies only slightly with conditions of evaporation. It is shown that a superheated vapour dryer is the most efficient thermally, but not in relation to rate of evaporation or productivity. The efficiency of many old stenters is greatly reduced by their housing in enclosures that are too large, badly insulated, and far from air-tight. Data are given for 12 stenters, showing the superiority of modern machines in relation to specific steam consumption (lb of steam per lb of water evaporated) and rate of evaporation (lb per sq. ft per h). The re-housing of an old machine on modern lines, reducing the amount of fresh air introduced during operation, more than halved the steam requirement whilst slightly increasing the rate of evaporation. The lowest efficiency on both counts was exhibited by a machine having no air recirculation. J.W.D.

### Improving Cotton Cord by Ozonisation

Kh. V. Usmanov, V. A. Kargin, B. I. Alkhodzhaev, and N. Sh. Inoyatov

Vysokomolekulyarnye Soedineniya, 2 (1960) 88–91

Chem. Abs., 54 (25 Aug 1960) 16841  
Medium-staple cotton cord was ozonised for 10 h and then washed with water at 25°C. Its strength was increased stepwise from 7.37 to 7.92 and to 7.95 kg/mm<sup>2</sup>. The fats and waxes were decomposed and completely washed off. The sorptive properties increase on ozonisation and decrease on washing and extraction because the low-molecular substances formed by ozonisation are more hydrophilic than cellulose. C.O.C.

### Micro-organism Growing on Cloth. VIII—Treatment of Cloth containing Cellulose Acetate with 8-Hydroxyquinoline Copper Acetate

K. Ichishima Kasei-gaku Zasshi, 11 (1960) 40–43

Chem. Abs., 54 (25 Aug 1960) 16840

The adverse effect on cellulose acetate of treatment with 8-hydroxyquinoline Cu acetate is much reduced if the bath is kept at <60°C (at which  $\beta$ -transformation of the fibre occurs). C.O.C.

### Replacing THPC in the APO-THPC Flame-resistant Finish

T. D. Miles, F. A. Hoffman, and A. Merola

Amer. Dyestuff Rep., 49 (22 Aug 1960) 596–599

Compounds other than tetrakis(hydroxymethyl)phosphonium chloride can be combined with tri-1-aziridinyl phosphine oxide (CH<sub>2</sub>CH<sub>2</sub>)<sub>3</sub>PO (APO) as a durable flame-retardant for cotton. They must be able to catalyse the polymerisation of APO. An infrared

method for screening possible catalysts is described. A combination of APO and diammonium hydrogen phosphate is cheaper than existing APO finishes and is easier to handle, as no offensive fumes are generated and the bath is more stable. C.O.C.

**Cotton Fabric with Wet Crease Recovery produced with Formaldehyde without excessive Strength Loss**  
W. A. Reeves, R. M. Perkins, and L. H. Chance

*Amer. Dyestuff Rep.*, 49 (5 Sept 1960) 639-644  
Cotton cloth treated with a soln. of formaldehyde (7.4%) and HCl (19.5%) has improved wet crease recovery, but little or no dry crease recovery. Treatment by soaking is better than by padding followed by piling or rolling, and the treatment is more effective on mercerised than on unmercerised cotton. There is a loss of ca. 40% in tear strength and breaking strength. A.J.

**Problem of Chlorine Retention in "Minimum-Iron" Finishing of Cotton Fabrics**

W. Rümens, N. Götz, and R. Zeidler

*Amer. Dyestuff Rep.*, 49 (3 Oct 1960) P 752-P 763  
English translation of the article in *Melliand Textilber.*, 41 (Mar 1960) 330-338, abstracted in *J.S.D.C.*, 76 (Nov 1960) 650. M.T.

**Shrink-resisting of Wool with Neutral Permanganate or Acid Bromate in Concentrated Sodium Chloride Solution**

J. R. McPhoe *Text. Research J.*, 30 (May 1960) 358-365

Experiments using neutral permanganate (4-6%  $\text{KMnO}_4$  on weight of wool) and acid bromate (1-2%  $\text{KBrO}_3$  on wt. of wool), respectively, in conc. brine. The bromate treatment resulted in a chlorination more readily controlled to yield even treatment than simple acid chlorination. Batch processes are carried out at 20-40°C with normal equipment, total processing times being 1-2 h. Each process yields shrink-resisting wool at relatively low cost with practically no adverse effect, although the bromate-treated wool needs final softening. The permanganate treatment requires no analytical control, is non-fuming, results in no dyeing troubles and neither causes yellowing nor alters the handle. 30 References. C.O.C.

**Epoxy Ester Coatings for the Metal Litho Industry**  
M. A. Glaser, G. K. Hughes, E. J. Bromstead, and K. V. Sbrabian

*F.A.T.I.P.E.C. 4<sup>th</sup> Congr.*, Lucerne (1957) 19-28

*Chem. Abs.*, 54 (25 Aug 1960) 16867

The principal drawback to the use of epoxy ester coatings in this industry has been their inadequate curing properties; this makes impossible flat-stacking of epoxy-ester-coated sheets after normal stoving. Various factors that might influence the curing rate were investigated. Co, the optimum drier from the standpoint of final film properties, caused stratification of the varnishes upon ageing, though this did not appear to affect drying. There is as yet no explanation for this effect, which did not occur with other common driers. Appreciable improvement in drying characteristics was obtained by adding amino-formaldehyde resins. Small amounts of rosin gave slightly improved drying, fabrication and pasteurisation. Epoxyster compositions prepared from fatty acids have slightly better drying properties than those from methyl esters of the same acids. Use of such catalysts as Al butoxide also seems to improve drying performance. C.O.C.

PATENTS

**Controlling the Application of Size to Yarn**

Leesona Holt

BP 847,615 (22 Jan 1958)

A warp-sizing machine in which the pump etc. are so arranged that the amount of size to be taken up by the warp can be predetermined by the operator. During operation, what is happening at any one point can be clearly seen. Simple mechanical adjustment ensures at all times an absolute accurate measurement of the size put on the warp. C.O.C.

**Aqueous Dispersions of Ultramarine (C.I. Pigment Blue 29) as Sighting Colours for Synthetic Polymer Fibres**

Deering Milliken Research Corp.

USP 2,920,975 (3 Nov 1955)

An aqueous dispersion of Ultramarine,  $\text{Na}_2\text{SO}_4$ ,  $\text{Na}_2\text{SiO}_3$ ,  $\text{NaHSO}_3$  or Na formate (25-75% on wt. of pigment) and

a fatty acid sodium soap (50-200% on wt. of pigment) is quite stable and is very satisfactory for the fugitive tinting of synthetic polymer yarns. C.O.C.

**Dimensional Stabilisation of Cellulosic Fabrics**

E. Robbart

BP 845,118 (15 Feb 1957)

The fabrics are exposed to organosilicon halide vapour, then treated with aqueous alkali and dried under tension. C.O.C.

**Crease-shedding Finish on Cellulosic Fabrics**

Sun Chemical Corp.

BP 845,468 (6 Mar 1958)

Use of an aqueous solution containing a 1-substituted-3,5-dimethylol-2,6-dihydrotriazin-4-one and an acidic catalyst, followed by drying and baking, yields a product which does not retain chlorine and has a soft handle. C.O.C.

**Crease-shedding Finish**

B. F. Goodrich Co.

BP 843,114 (U.S.A. 19 Sept 1955)

The material is treated with a melamine- or urea-formaldehyde precondensate and an elastomeric copolymer of a vinyl halide. The latter contains as internal plasticiser vinylidene chloride, alkyl acrylate (Alk of 6-10 C), an acrylamide, an acrylonitrile, isobutylene, acrylic acid, ethylene, 2-chloroethyl vinyl ether, isobutyl vinyl ketone and trichloroethylene. The ratio of resin to copolymer is from 25:75 to 55:45 and the amount of mixture applied is such as to deposit 5-20% solids on the material. Finally, the impregnated material is baked. The treated material is highly crease-shedding and its tearing, breaking, and bursting strengths are much higher than those of fabric treated with resin alone. The abrasion resistance is excellent and there is little tendency to pick up chlorine. C.O.C.

**Drip-dry Crease-shedding Finish on Cellulose**

Bleachers' Association

BP 846,287 (10 Mar 1958)

The fabric is treated in aqueous medium with a film-forming polyol, a cross-linking agent and a water-soluble polyester comprising structural units connected by ester linkages and which contains free acidic groups. Thus polyvinyl alcohol, 97% hydrolysed, (1 g) is dissolved in hot water (80 ml). When cool, tetramethylolacetate diurea (25 g) is added followed by a triethylene glycol-succinic acid (0.5:1.0 mol) condensate dissolved in water (20 ml) and 40% formalin (5 g). Cotton poplin is impregnated with the resulting solution, dried, and baked for 150 s at 130°C. The finished cloth has good wet crease-resistance and good fastness to washing and abrasion. C.O.C.

**Water-repellent Finish on Cellulose Fabric**

E. Robbart

BP 845,119 (15 Feb 1957)

The fabric is conditioned in an atmosphere of 30-90% r.h., then passed through an aerosol atmosphere of an organosilicon halide, treated with aqueous alkali and dried. C.O.C.

**Rendering Fibrous Materials Water-repellent**

ICI

BP 845,651 (7 Mar 1958)

The materials are treated with a methylpolysiloxane fluid in presence of an organotin oxide and a metal octoate and then are heat-cured. Thus wool and cotton gabardines were treated with a mixture of (a) Zn 2-ethyl hexoate and dibutyl-tin oxide dissolved in toluene, and (b) a white spirit solution of a mixture of methylpolysiloxane, one having 1.1  $\text{CH}_3$  groups per Si atom and the other 2.02. The treated cloths gave excellent Bundesmann tests. C.O.C.

**Increasing the Tensile Strength of Loose Wool and Wool Yarns**

Zschimmer & Schwartz Chemische Fabrik und Gerbstoffwerk Oberlahnstein

BP 845,136 (Germany 5 Jan 1957)

The wool is treated with silicon tetrachloride in non-aqueous solvent. Thus wool yarn was treated for 15 min at 20°C in a 4% solution of silicon tetrachloride in trichloroethylene. It was then squeezed out and dried for 20 min at 90°C. The treated yarn had tensile strength 1.3 g/den., compared with 0.85 for the untreated yarn. C.O.C.

**Water-repellent Finish**

FBy

BP 844,985 (Germany 26 July 1957)

Treatment with chromium complex salts and silicones has a synergistic action in producing a water-repellent finish. The compounds are applied in any sequence, if desired with intermediate drying, or simultaneously. Thus

a water-repellent finish fast to washing and dry cleaning is obtained on wool by treatment with an aqueous dispersion of an isopropyl alcohol solution of stearato-chromic chloride to which dispersion has been added a methylene chloride solution of hydrogenmethylpolysiloxane (viscosity 250 cS). Drying is done at 60–90°C. C.O.C.

#### Setting Polyethylene Fibres, Films, etc.

FH BP 849,670 (Germany 31 Dec 1955)

The shrinking capacity of stretched polyethylene filaments or film is reduced and their stress-strain behaviour stabilised by heating them at 80–50 deg. C below their m.p. for 30–1200 s. The treated materials are quite stable when subsequently heated to  $\geq 5$  deg. C below the temperature at which they have been treated. C.O.C.

#### Polyester Fibres Stabilised to Ultraviolet Radiation

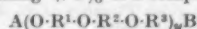
General Aniline USP 2,920,978 (26 May 1958)

Treatment with a 0.5–3.0% solution of a 2,2'-dihydroxy-4,4'-dialkoxybenzophenone or a 2,2',4-trihydroxy-4'-monoalkoxybenzophenone in diethylene glycol at 340–365°F for 5–15 s stabilises polyester fibres against degradation on exposure to ultraviolet radiation. When the treatment is done before dyeing, not only the fibre but also the dye is protected. Protection is obtained even with those dyes that otherwise accelerate the rate of degradation. C.O.C.

#### Reducing the Tendency to Pill of Fabrics containing Polyester Fibres

DuP USP 2,920,980 (28 Mar 1956)

A liquid containing  $\leq 5\%$  of a compound of formula—



( $R^1$ ,  $R^2$  and  $R^3$  = alkylene; A = H, Alk, Ar or acyl; B = OH, alkylether, arylother or an alkyl or arylother radical; y = value to give an alkylene oxide component of mol.wt.  $\leq 300$ ) is applied by padding, brushing or spraying and then one or both surfaces only of the fabric is heated rapidly to  $\leq 200^\circ\text{C}$ , care being taken to ensure that the interior of the fabric is kept at as low a temperature as possible. C.O.C.

#### Coating Polyester or Polyamide Fabrics with Vinyl Chloride Polymers or Copolymers

Imperial Chemical Industries of Australia and New Zealand BP 852,499 (Australia 12 Dec 1958)

The fabric is first coated with a composition containing a butadiene-acrylonitrile rubber, a vinyl chloride-vinyl acetate-vinyl alcohol copolymer, a chlorinated rubber and an organic triisocyanate. This acts as an anchoring coating for a subsequent coating of a vinyl chloride polymer or copolymer. The first coating is preferably plasticised with a polymeric glycol ester modified with a monocarboxylic acid, e.g. benzoic acid-modified polypropylene adipate, as this imparts improved tear strength to the final product. C.O.C.

#### Improving the Handle of Metal-coated Fibres

Owens-Corning Fiberglass Corp.

USP 2,920,981 (30 Mar 1954)

Metal-coated glass fibre yarn is treated with an aqueous composition containing rubber latex (0.3% by wt.), tetraethylene pentamine stearic acid condensate (0.15) and starch (0.1). C.O.C.

#### Ladder-resistant Hosiery

S. O. B. Ljungbo BP 847,126 (Sweden 30 June 1956)

Hosiery resistant to runs is obtained by applying a ladder pattern of spots of an adhesive comprising a polymer emulsion or solution and a solvent for the fibre. Passage of air through the stocking, during application, prevents closure of the fabric meshes and final heat treatment produces bonding at the yarn intersections. G.E.K.

#### Permeable Polymeric Coatings—Leathercloth

DuP BP 849,155 (U.S.A. 28 Jan 1957)

A polymer formed by chain-extending an isocyanate-group-terminated reaction product of a polyalkylene ether glycol and a di-isocyanate is dissolved in a hypodermic solvent. The substrate is coated with this solution and then exposed to an atmosphere of 15–100% r.h. at a dry bulb temperature of 50–100°F and then the residual solvent is removed. The solution is particularly suitable for coating cloth to produce leather-like products. C.O.C.

#### Coated Fabric having Antistatic Properties

Dunlop Rubber Co.

BP 853,186 (25 July 1956)

An antistatic fabric useful, e.g., for car upholstery, consists of a backing containing fibres incorporating an electrically conductive Carbon Black (C.I. Pigment Black 6 and 7) and a coating of natural or synthetic rubber or resin composition. C.O.C.

#### Decorations on Cloth or other Sheet Material

T. Brenna

BP 848,258 (Italy 27 July 1957)

A polymerisable adhesive is applied as a pattern to the cloth and is then polymerised. Apertures in a selected pattern are then formed in those parts of the cloth covered by the adhesive. Before the polymerisation, fibres or coloured powders can be dusted on to the adhesive coating. Suitable apparatus is described. C.O.C.

#### Antivesicant Finish

U.S. Secretary of War

USP 2,920,986 (13 Apr 1959)

USP 2,921,031 (30 May 1942)

Protection against mustard gas is given by fabric impregnated with a chloramide. Bis-2,4,6-trichlorophenyl chlorourea is particularly suitable because it readily goes into solution and is very stable on cloth exposed out of doors. C.O.C.

Hosiery Finishing Machine (I p. 27)

Warp Sizing Machine (I p. 28)

Tygaflor PTFE-coated Glass Fabrics and Yarns (VI p. 40)

Carbamoyl ethylation of Polyvinyl Alcohol Fibres with Acrylamide (VI p. 40)

Modified Manila Hemp (VI p. 40)

Rendering Synthetic Polymer Yarns Non-slipping (VI p. 40)

Improving the Water Absorption of Nylon (VI p. 41)

Simultaneous Dyeing and Resin Finishing of Textiles (VIII p. 43)

Cellular Surface Coatings having an Embossed Appearance (IX p. 44)

Electronics in the Textile Industry (XIV p. 51)

Wash-and-Wear Fabrics—Appearance after Home Laundering (Tentative Test Method 88–1960) (XIV p. 51)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

### The Mechanism of Pulp Resin Accumulation at Solid Surfaces

E. Back

*Svensk Papperstidning*, 63 (15 Sept 1960) 556–564

A description of model flow experiments in which unbleached sulphite pulps were stirred with various stirrers and baffles. The resin deposited on different parts of the apparatus was measured at intervals. If the deposition surface is porous, e.g. woollen felt, resin accumulates where deposited. If the surface is smooth the resin will flow along it. A.J.

### Colouring of Paperboard

M. J. Landbeig

*Pulp Paper Mag. Can.*, 61 (1960) T91–T96

*Chem. Abs.*, 54 (10 Aug 1960) 15929

A critical review of the dyes and methods used. C.O.C.

### Penetration of Varnishes and Inks into Paper under Pressure

R. R. Coupe and B. Hsu

*J. Oil & Col. Chem. Assoc.*, 43 (Oct 1960) 720–736

Penetration increases with initial film thickness, approaching a limiting value which depends upon impression time and pressure, and ink viscosity. The percentage transfer curve shows a max. which always precedes the limiting penetration; the discrepancy is due to the non-uniformity of the paper surface and pores. The penetration curve corresponds to a logarithmic normal distribution function. In the region of limiting penetration the amount of liquid actually penetrating into the paper can be calc. from the transfer data; it appears to be linearly related to the fall in reflectance. The amount of liquid required to fill the surface depressions of the paper is independent of pressure, time, and viscosity. For a paper of very open structure there is neither limiting penetration nor max. transfer. It is believed that, owing to the



compression and recovery of the paper, some pores filled with liquid have spaces in them. Initial penetration may occur in the wide pores, with subsequent re-distribution resulting in the pigments being concentrated in the wide pores and the vehicle in the narrow ones. J.W.D.

## PATENTS

### Preventing Reddening of Unbleached or Semi-bleached Sulphite Pulp

Canadian Industries BP 846,079 (Canada 11 July 1958)

Discoloration during bleaching is prevented by adding  $\leq 0.01\%$  of hydrazine, dimethyl hydrazine, hydroxylamine or K or Na borohydride to the pulp in the head box, or to the sheet by spraying before bleaching, or to the slurry before the paper machine to remove any reddening that has occurred. R.A.

### Improving the Freeness and Drainability of Pulp by Means of Surface-active Agents

Canadian Aniline and Extract Co.

BP 852,678 (Canada 18 Mar 1958)

A water-soluble condensate of an alkyl phenol, fatty alcohol, acid or amine, or a thioalcohol, all of 8–18 C and 7–25 molar proportions of ethylene oxide, is sprayed on to the pulp web. R.A.

### Inorganic Paper

General Aniline

BP 848,936 (U.S.A. 19 Dec 1955)

To a stock of glass, silica or asbestos fibres or mica is added 0.1–0.5% of a water-soluble polyvinyl pyrrolidone which is rendered insoluble by adding a vinyl methyl ether-maleic anhydride copolymer or by heat. R.A.

### Colophony Adducts as Paper-sizing Agents

British Bewoid Co.

BP 851,187 (Netherlands 19 Dec 1956)

Maleic acid or anhydride (5–25%) is treated with colophony (95–75%) for 20 min at  $\leq 120^\circ\text{C}$ . The resultant adduct is successively partly neutralised by NaOH, partly esterified with 0.5–10% by wt. of rosin of a half-etherified polyoxyethylene alcohol of  $\leq 18$  C, partly neutralised with 5–6% by wt. of rosin of triethanolamine, and finally completely neutralised with NaOH. R.A.

### Coloured Paper

ICI

BP 851,546 (23 Dec 1957)

Pigments are incorporated in the paper during its manufacture, using as assistant the condensate obtained by heating, in absence of water, a polyalkylene polyamine with cyanamide, a derivative thereof, or a derivative of cyanocyanamide, e.g. the condensate of phenylbiguanide and tetraethylenepentamine described in Example 1 of BP 798,061 (J.S.D.C., 74 (1958) 705). C.O.C.

### Use of Air Blast to produce Matt Finish Sheet by Dope Casting

Colanese Corp. of America USP 2,921,345 (15 Dec 1954)

When sheeting is produced by solvent casting, immediately the surface of the sheeting that has been in contact with the casting surface leaves that surface and while it still contains a considerable amount of solvent, it is treated with a stream of air. This results in a matt finish on that surface. C.O.C.

### Aminoplast-impregnated Surfacing Sheets

Th. Goldschmidt

BP 845,821 (5 Sept 1958)

Paper, textile and similar sheets for improving the surface of laminates, boards and plywood, are first impregnated with a resin having high flowability during the pressing operation. After drying, the sheet is coated with a soln. of a rapidly drying hardening resin having a lower degree of flowability during pressing. The degree of flowability is controlled by the degree of condensation or by the addition of hardening retarders or accelerators. R.A.

### Coated Paper

Minnesota Mining and Manufacturing Co.

BP 850,818 (U.S.A. 13 Jan 1956)

An unsized porous paper is coated on one side with a lipophobic fluorocarbon sizing agent, e.g. a Cr complex of a fluorocarbon monocarboxylic acid, and on the other with a hot-melt thermoplastic coating composition, e.g. wax, asphalt or a waxy carbon ink coating. The papers produced find use as wrappers, liners, adhesive sheets and carbon papers. R.A.

### Coating Sheets of Paper

T. B. Bush and H. N. Bush BP 852,489 (4 June 1958)

The sheets are fed separately between a backing cylinder and an applying roller, with the latter rotating in the reverse direction at the same or higher speed than the cylinder. R.A.

### Non-adherent Cellulosic Materials

General Electric Co. BP 852,717 (U.S.A. 26 Mar 1958)

Paper and paperboard will not adhere to asphalts, bitumen, tars, waxes, paraffin solids, flour-containing pastes, frozen foods and high-mol.-wt. polymers if treated with an emulsion containing a linear polydimethylsiloxane containing terminal Si-bonded OH-groups (2–6 parts by wt.), a methyl-polyaloxane resin having 1.05–1.4 methyl groups per Si-atom (1), water (3–75), 0.05–10% tin as dibutyl tin dilaurate, based on the weight of linear polydimethyl siloxane, and 0.1–3% polyvinyl alcohol, as emulsifier, based on the total weight. R.A.

### Carbon Paper producing Copies for Thermal Copying

Caribonum

BP 849,756 (30 Apr 1958)

Modification of BP 840,673. The base sheet is coated with a fluid containing a dispersion of Graphite (C.I. Pigment Black 10), a vinyl chloride-vinyl acetate copolymer dissolved in a volatile solvent. The Graphite is dispersed in a liquid which is a non-solvent for the copolymer, e.g. ethanol. The fluid may contain additional colorants. The product yields copies suitable both for thermal copying and for ordinary purposes. C.O.C.

Calender for High-gloss Finishes (I p. 28)

Manifolding Sheet Material (IX p. 44)

Colouring of Paperboard (XI p. 47)

Matt Finish on Plastic Sheet (XIII p. 49)

Polishing or Imparting a Pattern to Thermoplastic Polymeric Plastic Films or Coatings (XIII p. 50)

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

## PATENTS

### Tanning

A/S Toten Cellulosefabrik

BP 849,876 (Norway 19 Mar 1958)

Treating the skins with concentrated cellulose extract, e.g. Totannin (ammonium bisulphite lye), before vegetable tanning much shortens the time required for tanning without detracting from the quality of the resultant leather. C.O.C.

### Fat-liquoring

R. E. Ushakoff

BP 852,986 (13 Jan 1959)

The fattening agent is first supplied to the skin in the required amount and then distributed throughout the skin by subsequent processing. This renders it unnecessary to keep the skin in presence of the fat liquor for the time required for impregnation or treatment with excess of fat liquor. Thus a skin solvent-tanned by the process of BP 755,969 (J.S.D.C., 72 (1956) 555) or solvent-dehydrated by the process of BP 807,741 is coated with the fattening agent. The skin is then dried with the result that the fattening agent replaces the solvent throughout the skin. C.O.C.

### Waterproofing Leather

British Leather Manufacturers' Research Assocn.

BP 848,838 (7 May 1957)

When leather is treated with an organic-solvent solution of a silicone, migration of the silicone during drying out of the solvent is greatly reduced if the silicone solution is made viscous by adding a higher elastomeric polymer or rubber-like substance. This greatly improves the waterproof finish. C.O.C.

## XIII—RUBBER; RESINS; PLASTICS

### Effect of Driers on Drying Times of Clear and Pigmented Alkyd Resins

L. Dintenfuss

J. Oil & Col. Chem. Assocn., 43 (Oct 1960) 709–719

Drying times of alkyd resins vary with composition of drier and duration of storage. For any one drier combination, the drying time may increase or decrease.



Addition of pigments such as titania affects drying time through the adsorption of driers from the liquid phase; only non-adsorbed drier is active in the drying process. There are conditions under which Mn, Co-Mn, and Mn-Pb driers become more effective following the adsorption resulting from pigment addition. The retarding influence of Mn driers remains unexplained. The adsorption of driers may be reduced by the addition of inactive soaps such as Mg or Ca naphthenates. Metal soaps such as Fe naphthenate affect the viscosity of alkyd resin soln., an increase usually resulting in direct proportion to the quantity of soap or drier added. Micellar and colloidal phenomena, in addition to chemical factors, may be involved in the drying process but the mechanism is as yet neither explained nor fully understood. J.W.D.

#### Colours for Plastic Floorings

C. Musgrave *Fibres & Plastics*, 21 (Oct 1960) 291-294

Plastic floorings embrace rigid and semi-rigid vinyl tiles, flexible vinyl tiles and continuous flooring, vinyl-coated felt, coumarone-indene tiles, linoleum, and tufted carpets having rubber or plastic backing. The properties required in pigments for use in materials from each of these categories are discussed in relation to both manufacture and service conditions. The colorants generally used in floorings are classified chemically and their properties and limitations are shown in a table. A further table embodies specific recommendations of pigment types for each type of flooring. J.W.D.

#### Ageing of Plastics

P. Dubois and J. Hennicker

*Plastics*, 25 (Oct 1960) 428-430

The presence of amorphous or cryst. regions, their juxtaposition within the structure, and the presence of internal stresses are all forms of anisotropy which promote ageing. These factors are discussed in relation to individual types of macromolecular structure; additives such as pigments and plasticisers also have a marked effect. Residual reactivity (especially at the ends of chains) may lead to modifications if reaction is promoted by favourable conditions, e.g. heat. The effect of structural bond energy is discussed briefly. External factors promoting ageing are considered under these headings: photochemical influences, thermal stress (including thermal shock), chemical stress (oxidation, effect of moisture, etc.), and mechanical stress leading to early rupture or other failure. 12 References. J.W.D.

#### Biological Deterioration of Plastics

H. J. Huock *Plastics*, 25 (Oct 1960) 419-422

A review of present knowledge of the liability of plastics (including additives such as plasticisers and fillers) to attack by micro-organisms, insects, and rodents. Vulnerability of mixtures of natural and synthetic fibres is also discussed. There is a valuable list of references. J.W.D.

#### PATENTS

##### Improving the Affinity for Dyes of Polymers or Copolymers of Ethylene and Propylene with Simultaneous Imparting of Lyophilic and Antistatic Properties

Montecatini Società Generale per l'Industria Mineraria e Chimica BP 850,471 (Italy 1 June 1956)

The resins described in BP 843,190 have greater affinity for acid dyes if the polymer grafted on to the initial polymer contains in the polymer chain an acidic or a basic nitrogenous group or is convertible by reaction with organic basic nitrogenous compounds or by hydrolysis to yield a group having affinity for acid dyes. C.O.C.

##### Pigmented Polyolefin Composition

Godfrey L. Cabot BP 852,371 (U.S.A. 1 Feb 1957)

A polyolefin moulding composition which can be crosslinked by heating, comprises a solid thermoplastic polyolefin resin (100 parts by wt.), an organic peroxide (<0.5) and a neutral to alkaline, solid filler (10). The filler should be in colloidal state and when alone with the resin should not yield a composition that bubbles at < 500°F. C.O.C.

##### Removing Monomers from Polyamides

Courtauld BP 853,330 (12 July 1957)

The distillate from a molten mixture of a polyamide and its monomer, particularly nylon 6 and caprolactam

is passed first through a condenser maintained at > m.p. of caprolactam and then through a cold condenser. A considerable proportion of the caprolactam is obtained as a liquid in the first condenser and the remainder as a solid in the second condenser. C.O.C.

##### Dyed Polyester Resins

Dr. Kurt Herberts & Co.

BP 852,646 (Germany 18 Dec 1956)

Dyed polyester resins of very good fastness properties are obtained if dyes having carboxylic acid, amino and/or hydroxyl groups are present in the reaction mixture used to form the resin and if resin formation is carried out under conditions to ensure that the dye is chemically incorporated into the resin molecule. C.O.C.

##### Polymeric Organo-Boron Phosphorus-containing Compounds

United States Borax & Chemical Corp.

BP 848,656 (U.S.A. 3 July 1958)

Phenylphosphino-phenylborane polymers having as the sole recurring structural unit  $PC_6H_5-BC_6H_5$  bonded together only through B-P bonds are used to prepare films, shaped objects, etc. They are prepared by condensing phenylphosphine and phenylboron dichloride. C.O.C.

##### Sheeting containing Pearlescent Pigments

United States Rubber Co. USP 2,291,346 (31 Dec 1957)

A carrier is given a fluid coating of a resinous material contained flaked pigment. The carrier is then folded into a web of U-type cross section having the coating within its trough and is then passed between suitable platens. The pressure from the platens causes the coating material to flow transversely and so orient the pigment flakes in the direction of flow, i.e. in the plane of the sheeting to be formed. Immediately the pigment is orientated the coating material is gelled by passing the carrier between heated platens aligned with the pressure platens. C.O.C.

##### Leather-like Products

Wacker-Chemie BP 847,837 (Germany 16 Mar 1956)

A silicone-rubber composition vulcanising at room temperature is applied to a flexible support. The leather-like products produced retain their elasticity over the temperature range 200°C to -70°C and have a high degree of soil resistance. In addition they are fast to water, weathering and light. C.O.C.

##### Improving the Colour Stability of Polyurethane Cellular Products

C. L. Wilson USP 2,921,866 (9 Oct 1956)

The rate of discoloration or yellowing of polyurethane foams is greatly retarded by treating the finished foam with HCHO, keten, acetic anhydride, ethylene oxide or monoisocyanates. Particularly good results are obtained by use of a monoisocyanate either in solution or as a vapour. C.O.C.

##### Rendering Thermoplastic Articles Heat-, Stain-, and Scratch-resistant

Union Carbide Corp. USP 2,921,870 (23 Apr 1957)

Treatment with organo halogenosilanes so as to cause penetration of at least 3 mils and to impart definite opacity results in marked increase in resistance to staining, e.g. by fruit or vegetable juices, and also to heat and scratching. C.O.C.

##### Embossing Thermoplastic Materials

W. Baulcombe and A. C. Cartwright

BP 848,239 (22 Oct 1957)

Polythene or plastic having similar properties has a mask placed on its surface to protect those parts which are not to be embossed. Molten or softened plastic is then applied to the parts to be embossed, followed by heat and pressure. Finally the mask is removed. C.O.C.

##### Matt Finish on Plastic Sheeting

Lawes Bros. and Abrasive Developments

BP 851,021 (25 Apr 1957)

An aqueous slurry of abrasive particles is thrown by centrifugal action on to the surface of the sheet.

BP 851,022 (25 Apr 1957)

The deposit of powdered plastic left on the sheet can cause marking of the film when it is handled. This is overcome by giving a thin coating of a film-forming material. C.O.C.

### Polishing or Imparting a Pattern to Thermoplastic Polymeric Plastic Films or Coatings

International Polaroid Corp.

BP 850,553 (U.S.A. 1 July 1957)

A softening agent is applied to the surface of the film which is then immediately brought into contact with a hot surface and held against it until the film's surface has been re-hardened and polished or patterned.

C.O.C.

Agglomerating Powdered Pigments (IV p. 38)  
Dispersing Powders in Non-aqueous Liquids (V p. 39)  
Improving the Adhesion of Printing Inks to Plastics (IX p. 45)  
Setting Polyethylene Fibres, Films, etc. (X p. 47)  
Permeable Polymeric Coatings—Leathercloth (X p. 47)  
Use of Air Blast to produce Matt Finish Sheetting by Dope Casting (XI p. 48)  
Sheeting containing Pearlescent Pigments (XIII p. 49)  
Rendering Thermoplastic Articles Heat-, Stain-, and Scratch-resistant (XIII p. 49)  
Analysis and Testing of Plastics (XIV p. 51)  
Infrared Dichroism and Crystallinity in Polyethylene and Polyethylene Suberate (XIV p. 51)

## XIV—ANALYSIS; TESTING; APPARATUS

### Diazotised *p*-Nitroaniline as Location Agent for Ammonium Salts of Aliphatic Acids

A. E. Whitfield *J. Chromatography*, 4 (Oct 1960) 350-351

Paper-chromatographic spots of the above salts may be detected by diazotised *p*-nitroaniline spray followed immediately by a spray of 15% (wt./vol.) aq. sodium carbonate. Ammonium salts of  $C_1$ - $C_8$  fatty acids,  $\alpha$ -hydroxyisocaproic,  $\alpha$ -hydroxyisovaleric,  $\alpha$ -ketoisocaproic, malic, lactic, succinic, citric, tartaric, glycollic, tiglic, dimethacrylic and methacrylic acids have been detected.

F.J.

### $R_f$ Values of Some Anthocyanins and Anthocyanin Intermediate Glycosides

*J. Chromatography*, 4 (Oct 1960) D20

$R_f$  values of some anthocyanins and some of their related 5-glucosides are listed from original data of J. B. Harbourn, *Biochem. J.*, 74 (1960) 262.

F.J.

### $R_f$ Values of Some Flavonols

*J. Chromatography*, 4 (Oct 1960) D22

$R_f$  values of quercetin, kaempferol and their 3-glucosides and glucose are given for different solvent systems abstracted from data by V. Kwasniewski, *Planta Med.*, 7 (1959) 35.

F.J.

### Stable Diazo Salts for Chromatographic Spray Reagents

I. A. Pearl and P. F. McCoy

*Anal. Chem.*, 32 (Oct 1960) 1407-1410

Aqueous solutions of 30 different commercial stabilised diazonium salts are tested for location and identification of phenolic compounds and aromatic amines on paper chromatograms. 20 Phenolic compounds, related to wood chemistry, are used as test material and the colours produced with these before and after spraying with astd. sodium carbonate solution are used for identification.

P.B.S.

### Chromatography of Technical Organic Dyes. I.—Capillary Analysis and Column-adsorption Chromatography

L. Roosen

*Mededel. Vlaam. Chem. Ver.*, 21 (1959) 138-239 (in German)

*Chem. Abs.*, 54 (25 Aug 1960) 16833

Review, 494 references.

C.O.C.

### Investigation of Dyes in Foods, Beverages, and Medicinal Drugs by Paper Chromatography

H. S. Dellinger *Anal. Biomol.* (Madrid), 12 (1960) 9-18

*Chem. Abs.*, 54 (25 Aug 1960) 16681

The advantages and limitations of the extraction and concentration methods for different dyes are discussed. Adsorption on Brockman's  $Al_2O_3$  and on wool are the most recommended methods. Ascending paper chromatography gave the best results, the dyes being identified by their  $R_f$  values, reaction to ultraviolet radiation, and change in colour when treated with NaOH or HCl. Acid and basic dyes were developed with Na citrate (2 g) in

5% (wt./vol.)  $NH_4OH$  (50 ml) for 1.5-2.5 h. Liposoluble dyes after extraction were spotted in paper previously impregnated with kerosene and developed for 1.5 h in methanol-acetic acid (95:5 vol./vol.).

C.O.C.

### Analytical Determination of Triphenylmethane Dyes by Bromination

M. Matrká

*Chem. průmysl*, 8 (1958) 583-585

*Chem. Abs.*, 54 (25 Aug 1960) 16835

Comparison of the determination of triphenylmethane dyes by the reductometric method with 0.1-N VSO<sub>3</sub> with potentiometric titration with a Pt electrode with 0.2-N  $KBrO_3$  (KBr). The dye (0.1 g) is dissolved in N HCl (100 ml) and titrated. The method can be used to determine the pure dye, but the leuco bases were also brominated. The method is suitable for determining the leuco bases.

C.O.C.

### Paper Chromatography of Some U.S. Certified Dyes

F. J. Bandelin and J. V. Tuschhoff

*J. Amer. Pharm. Assoc.*, 49 (1960) 302-304

*Chem. Abs.*, 54 (25 Aug 1960) 16748

Most of the commonly used U.S. certified dyes can be separated and identified by paper chromatography by using a single-solvent system of 2% aq.  $NH_4OH$  containing 2% isobutyl alcohol.

C.O.C.

### Rapid Analysis for Quality Control of Paint Driers

T. McCourt and H. Tuberg

*Australian Paint J.*, 4 (11) (1960) 15-17

*Chem. Abs.*, 54 (25 Aug 1960) 16862

Titration with the disodium salt of ethylenediaminetetra-acetic acid provide a general technique for analysis of Pb, Co, Mn, Zn and Cd. Few reagents are required and the apparatus is simple and the reagents stable.

C.O.C.

### Evaluation of Mill-methods for Identifying Cotton Degradation

A. Arachian and E. Stefan

*Ind. textila* (Bucharest), 10 (1959) 426-432

*Chem. Abs.*, 54 (25 Aug 1960) 16841

Tests with several methods made on cotton cloth impregnated with various amounts of NaOCl and then air dried at 110°C and ironed at 150°C showed that treatment in 15% NaOH (Willow-Markert) is very useful as it rapidly distinguishes between mechanical and chemical damage and can be used on all cottons except those dyed with Aniline Black (C.I. Oxidation Base 1). The Nessler reagent test should be done in absence of light. Methylene Blue (C.I. Basic Blue 9) reactions as described in the literature were observed only at  $[H_2SO_4] > 10$  g/l. and  $> 2$  g/l. active Cl and are deemed practically useless. KI-starch is the most useful Cl indicator, detecting as little as 0.01 g/l. Cl. No reagent permitted distinction between oxidative and hydrolytic degradation.

C.O.C.

### Quantitative Separation of Wool and Cellulose Hydrate Fibres—II

M. Kehren and C. Maurer

*Z. ges. Textil-Ind.*, 62 (1960) 19-22

*Chem. Abs.*, 54 (25 Aug 1960) 16838

As a result of comparative tests Druce's method (*Chem. Abs.*, 50 (1956) 10414) proved suitable for quantitatively separating wool and cellulose hydrate fibres when the method was modified as follows. When testing fabrics the sample should be  $< 3$  g and for a patterned fabric 5 g, containing at least one repeat. To completely destroy the wool, the ratio of NaOCl solution to fabric must be 120:1 (ml/g). Preliminary extraction with distilled water is advisable. After the preliminary treatment, heavily milled cloth must be separated into warp and weft, tightly twisted yarns should be untwisted. When destroying the wool in milled cloth the reaction temperature can be 35°C at the start. The loss in weight of cellulose hydrate fibre samples in the blank test with x-NaOCl was always greater than that found by foreign authors. The NaOCl treatment of rayon staple and Cupramol fibres, in presence of wool, resulted in considerably less loss in weight; this agrees with the low values reported by other workers. In addition, reduction in strength of the NaOCl from 0.8 to 0.5 N resulted in less loss in weight, so that connection exists between the concentration of the oxidising agent and its action on cellulose hydrate fibres.

C.O.C.

### Estimation of Basic Groups in Wool by Dye-uptake Measurements

J. A. McClaren

*Arch. Biochem. Biophys.*, **86** (1960) 175-178  
*Chem. Abs.*, **54** (10 Aug 1960) 15945

Rate of absorption of acid dyes, e.g. Orange II and Orange G (C.I. Acid Orange 7 and 10, respectively) (as the free acids not Na salts) by wool from aqueous solution is markedly increased by presence of H-bond-breaking reagents. Wool absorbs Orange II acid very rapidly from solutions in anhydrous HCOOH. At concentrations  $> 3$  meq/l. the absorption is constant at 0.83 meq/g degreased wool (64s Merino); this corresponds to 1 equiv. of dye per basic group. The method can be used for estimating the basic groups in chemically modified wool; thus an over-carbonised wool absorbed only 0.6 meq/g, a dinitrophenylated sample 0.55, and an acetylated sample 0.12. C.O.C.

### Determination of Fatty Material in Wool Tops

M. van Overbeke, P. Outreman, and G. Mazingue

*Rev. Textile*, **15** (11) (1959) 15-21

Samples of top were impregnated with 3% of various oils and detergents, allowed to stand for several days, dried at 105°C for 2 h, extracted with ether and then with alcohol. The amounts extracted were (total extract as % of wt. of wool, % extracted by ether and % extracted by alcohol): arachis oil 3.17, 92.4, 7.6; mineral oil emulsified with a non-ionic surfactant 2.69, 100, 0; olein 3.13, 91.7, 8.3; soap 1.73, 3.4, 96.6; dodecylsulphate (30% active material) 1.99, 71.8, 28.2; an ethylene oxide-oleyl alcohol condensate 3.19, 88.7, 11.3. Ether extracted more material from fully conditioned samples that had not been dried than from those that had, because some fatty matter is lost during drying and there was partial hydrolysis of the material extracted. Alcohol extracted all the fatty matter in 4 h; it also extracted a small amount of proteinaceous material, as shown by decrease in the total S and cystine contents of the wool. Extraction with denatured alcohol in presence of air reddens the wool; this does not occur with pure alcohol.

*Ibid.* 32-35*Chem. Abs.*, **54** (25 Aug 1960) 16842

Comparison of the amounts of grease extracted from four types of wool material by ether,  $\text{CH}_2\text{Cl}_2$ ,  $\text{CHCl}_3$ ,  $\text{CCl}_4$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CCl}_4$  or acetone in each case followed by extraction with alcohol, shows that they were nearly constant for all systems.  $\text{CCl}_4$  gave results nearest to those obtained with ether;  $\text{CH}_2\text{Cl}_2$  extracted oxidised grease most effectively. C.O.C.

### Quantitative Chemical Analysis of Binary Mixtures of Acrylic and certain other Fibres (T.T.S. No. 63)

*J. Textile Inst.*, **51** (Sept 1960) P506-P570

The method applies to binary mixtures of acrylic fibres with wool, silk, cotton, polyamide, polyester and viscose rayon fibres, but not if the acrylic fibres are dyed with premetallised dyes. The acrylic fibres are dissolved in dimethylformamide at 60°C, and from the weight of residue, corrected where necessary for loss of the insoluble component in the specified pretreatment, the proportions of the two fibres in the mixture are calculated. M.T.

### Fibre Identification—A Scheme with emphasis on new Acrylic, Polyamide, Polyester and Cellulosic Fibres

R. S. Merkel

*Amer. Dyestuff Rep.*, **49** (5 Sept 1960) 625-637

A scheme of identification based mainly on fibre solubility and to a lesser extent on staining tests and microscopic appearance. 36 Tests are listed. An unknown fibre is not subjected to each test in turn, but the result of any one test determines the next test to be carried out. The scheme is also presented in tabular form, and there are appendices giving the relative solubilities and staining properties of cellulose acetates, acrylic, cellulosic, polyamide and polyester fibres. A.J.

### Recommended Method for Assessment of Light Fastness of Pigmented Surfaces and Systems

*J. Oil Col. Chem. Assoc.*, **43** (Aug 1960) 586-593

The Association's Council has agreed that steps should be taken to encourage adoption of the procedure of BS 1006:1955 for assessment of light fastness in the paints, printing ink, and allied industries. This paper

describes modifications which are advantageous in the use of BS 1006 in the pigment-using industries. The earlier work of the Association in this field is recorded and full acknowledgment is made that the Association is simply building on the vast amount of work done by the S.D.C. in the assessment of the fastness to light of dyed fabrics. C.O.C.

### Electronics in the Textile Industry

*Fibres & Plastics*, **21** (Oct 1960) 311-313

Account of electronic techniques and instruments for the control of yarn irregularity, moisture content—particularly important in the preparation of yarns for sizing—and temperature scanning in the heater zones of a machine for the balking of synthetic yarns. J.W.D.

### Creasability of Fabrics—Evaluation by Method of Residual Creasing Angle

T. Belleli

*Bull. Inst. Text. France* (87) (Mar-Apr 1960) 97-106

A recommended method is put forward on the basis of inter-laboratory trials. A scheme for rapid testing is described. J.C.F.

### Wash-and-Wear Fabrics—Appearance after Home Laundering (Tentative Test Method 88-1960)

A.A.T.C.C. Committee RA61

*Amer. Dyestuff Rep.*, **49** (3 Oct 1960) P746-P747

A revision of method 88-1958 to provide an alternative evaluation procedure (using three-dimensional plastic replicas) and to include fabrics having special surface effects. Method evaluates retention of original smooth or surface-textured appearance of fabrics after repeated home laundering. M.T.

### Resistance of Textile Fibres to Weathering

J. Lünenschloss and H. Stegherr

*Textil Praxis*, **15** (Sept 1960) 931-939

Account of the work done by the German Textile Research Institute (Reutlingen-Stuttgart). All the more important fibres were examined. Samples were supported on varnished steel frames 60 cm wide, exposed unprotected to atmosphere for one year, inclined at 45° and facing south. Records were kept of temperature, hours of sunshine, etc., and samples were examined and tested monthly for tensile strength, extension, work modulus, resistance to abrasion and knotting strength. Details are given of changes in tensile strength and extension (24 diagrams). Of natural cellulosic fibres, hemp is best. Of synthetic fibres, polyamide and polyester fibres are comparable but polyacrylonitrile and polyvinyl are best. L.A.T.

### Analysis and Testing of Plastics

*G. C. Ives Fibres & Plastics*, **21** (Oct 1960) 307-310, 313

A review of recently published methods and findings, with 32 references. J.W.D.

### Submicro-methods for the Analysis of Organic Compounds. XII—Determination of Carboxyl Group

R. Belcher, L. Serrano-Berges, and T. S. West

*J.C.S.*, (Oct 1960) 3830-3834

Details are given of a direct potentiometric titration method in aq. alcohol for the estimation of the carboxyl group using a modified glass/silver-silver chloride electrode system. The main problems were interference by atm.  $\text{CO}_2$ , overcome by working in nitrogen, and accurate location of end-point, achieved by using visual back titration with a mixed indicator of  $\alpha$ -naphtholphthalein-phenolphthalein (1:3) which changes from pale rose to pale green at pH 8.6 and to violet at pH 9. Data are given for the following acids: oxalic, succinic, tartaric, citric, hippuric, p-chlorobenzoic, m-trifluoromethylbenzoic, salicylic, phthalic, sulphonic, cinnamic, and potassium hydrogen tetrachlorophthalate. Benzoic acid is satisfactory as a primary standard, but, owing to volatilisation, it is essential to titrate it immediately after the weighing. H.H.H.

### Infrared Dichroism and Crystallinity in Polyethylene and Polyethylene Suberate

E. M. Bradbury, A. Elliott, and R. D. B. Fraser

*Trans. Faraday Soc.*, **56** (Aug 1960) 1117-1124

The spectrum of oriented films of polyethylene suberate has been observed with polarised infrared radiation.



Striking differences have been found between the spectra of amorphous and crystalline regions; bands near  $730\text{ cm}^{-1}$  are quite unsuitable for estimating the amorphous fraction in polyesters and polyethylene.

W.R.M.

### Simple Test for Detecting Differences Among Colour Matchers

K. McLaren

J.S.D.C., 76 (July 1960) 434-435

### Energy of Aqueous Wetting of Bulk Surfaces

P. E. Berghauer, R. J. Good, et al.

PB Report 151,593, 57 pp.

U.S. Govt. Research Repts., 31 (1959) 411

Chem. Abs., 54 (25 June 1960) 11682

The aqueous heats of immersion of polyvinyl chloride, polystyrene; polyacrylonitrile and of an acrylonitrile-vinylchloride copolymer were measured calorimetrically. The heats of immersion of polyvinyl chloride (degree of purity not known) and polystyrene are positive, i.e. heat is absorbed on immersion. This positive heat of immersion is caused by increase in entropy of water when adsorbed on the surface. Polyacrylonitrile has a large negative heat of immersion caused by H-bonding between it and water. The copolymer also has a negative heat of immersion, but smaller than that of polyacrylonitrile.

C.O.C.

### PATENTS

#### Indicator Papers Containing 2,4-Dinitrobenzeneazobenzene Sulphonic Acids

H. Wenker

USP 2,915,373 (20 July 1959)

Test papers containing the above dyes rapidly lose them into the test solution or, when they are treated with a test drop, the rapid spreading of the drop carries away some of the dye, leaving a whitened spot. These difficulties are overcome by adding to the dyes high-molecular-weight organic bases. Thus filter paper is impregnated with a mixture of the dye 2,4-dinitro-6-sulphobenzeneazo-1-naphthol (1 part) and the quaternary ammonium base (1.3) obtained from equimolecular amounts of ricinoleic acid, NN-diethyl-1,3-propanediamine and dimethylsulphate. The paper is green, becoming blue at pH  $< 7$  and yellow at pH  $> 5.5$ .

C.O.C.

#### Methods and Apparatus for Measuring Gel Strength and Viscosity

British Rubber Producers' Research Assn.

BP 842,977 (14 June 1956)

#### Viscosity Meter for Thixotropic or other Paints

BP 839,541 (8 May 1956)

#### Viscosity and Concentration Measuring Instruments

K. T. Kalle

BP 852,197 (Sweden 22 Nov 1957)

#### Apparatus for Retaining the Extensibility of Yarn

Dunlop Rubber Co.

BP 841,285 (15 May 1956)

Apparatus for carrying out extensibility tests rapidly on portions of a long length of yarn without damaging it.

C.O.C.

#### Testing the Crease Resistance of Cloth

Tootal Broadhurst Lee Co.

BP 846,875 (19 Dec 1955)

A sample of the fabric is folded in S or Z form and placed under pressure. After being pressed for a suitable time the sample is laid upon a flat surface and the inclination to the surface of a band of the fabric limited by the two parallel creases imposed on it by the pressure is measured.

C.O.C.

Absorption Spectra of Methylene Blue Adsorbed on Homoionic Bentonites suspended in Water (IV p. 31)  
Iodine Sorption on Cellulose Fibres (VI p. 39)

## XV— MISCELLANEOUS

### Reports on Definitive Rules for Nomenclature

J. Amer. Chem. Soc., 82 (5 Nov 1960) 5517-5584

Reports of commissions of the International Union of Pure and Applied Chemistry (IUPAC) concerning symbols, terminology, and nomenclature are given under four headings—(I) physical chemistry, (II) inorganic chemistry, (III) organic chemistry, (IV) amino acids, steroids, vitamins, and carotenoids. Comments concerning conflicts between

these recommendations and American practice and the rules of *Chemical Abstracts* have been interpolated in the text. Section I includes rules for chemical elements and nuclides, sign convention for e.m.f. and electrode potentials, the quantity pH and symbols and usages in physical chemistry and reaction kinetics. In Section II nomenclature rules are concerned with elements, compounds in general, ions and radicals, crystalline phases, acids, salts, co-ordination compounds, and polymorphism. Section III includes nomenclature rules for acyclic, monocyclic, fused polycyclic, spiro, terpene, and bridged hydrocarbons. Hydrocarbon ring assemblies and cyclic hydrocarbons with side chains are also discussed, together with heterocyclic systems. It is recognised that the recommendations of the IUPAC must involve compromises between these rules and those of various national organisations. However IUPAC rules may give guidance in seeking a wider measure of international agreement and also warning of instances where existing diversities may cause misunderstandings.

F.J.

### Bibliography on Molecular and Crystal Structure Models

D. K. Smith

NBS Monograph 14. U.S. Dept. of Commerce, National Bureau of Standards (20 May 1960) pp. 7

Brief descriptions of many of the models of crystal and molecular structure which have appeared in the literature is followed by a bibliography of the publications, grouped according to model types, which describe the models in more detail.

C.O.C.

### Factors Considered in Placement of Technical People

A. F. Hartford

Amer. Dyestuff Rep., 49 (11 July 1960) 501-503

An account of the procedure followed by the Colleges Relations Section of the du Pont company in judging the abilities of students.

C.O.C.

### Production Planning in a Vertical Textile Undertaking

W. Menzel

Textil Praxis, 15 (Sept 1960) 952-957

In addition to competitive price and high quality of merchandise, prompt deliveries on appointed dates contribute considerably towards reputation and goodwill which the manufacturer enjoys amongst his customers. It is the function of planning personnel to synchronise various stages of production from the supply of starting materials to the dispatch of finished products. Maximum machine utilisation, smooth flow of production, and absence of bottlenecks are important features of good production planning. The author discusses successive stages in production, and illustrates his ideas with flow charts and other visual aids.

L.A.T.

### Plant Maintenance and Working Conditions—I

F. H. Slade

Text. Manuf., 86 (Oct 1960) 397-400

Factors affecting machinery efficiencies and operative comfort include adequate control over temperature, air changes, and air filtration. Thus with drying units, air motion is of importance since the rate of air movement controls the humidity of the leaving air. Temperature and humidity control both the rate of drying and the final state of the dried material. A leak-proof and adequately lagged unit is required to avoid excessive heating costs. With air filtration units, the choice of filter must be suitable for the dust it is to intercept, and the period between servings must be such that the unit does not become choked. In refrigeration units the operating efficiency depends on adequate maintenance.

S.B.D.

### PATENT

#### Chemically Bonded Coatings on Aluminium

Amchem Products

BP 851,027 (14 Apr 1959)

Modification of BP 832,485 (J.S.D.C., 76 (July 1960) 450). The Na and K are present in the solution in the molecular ratio 1:1.9-2.1. This produces the most consistently satisfactory coatings and a solution having minimum tendency to supersaturation on heavy bath loadings.

C.O.C.

Energy of Aqueous Wetting of Bulk Surfaces (XIV this page)



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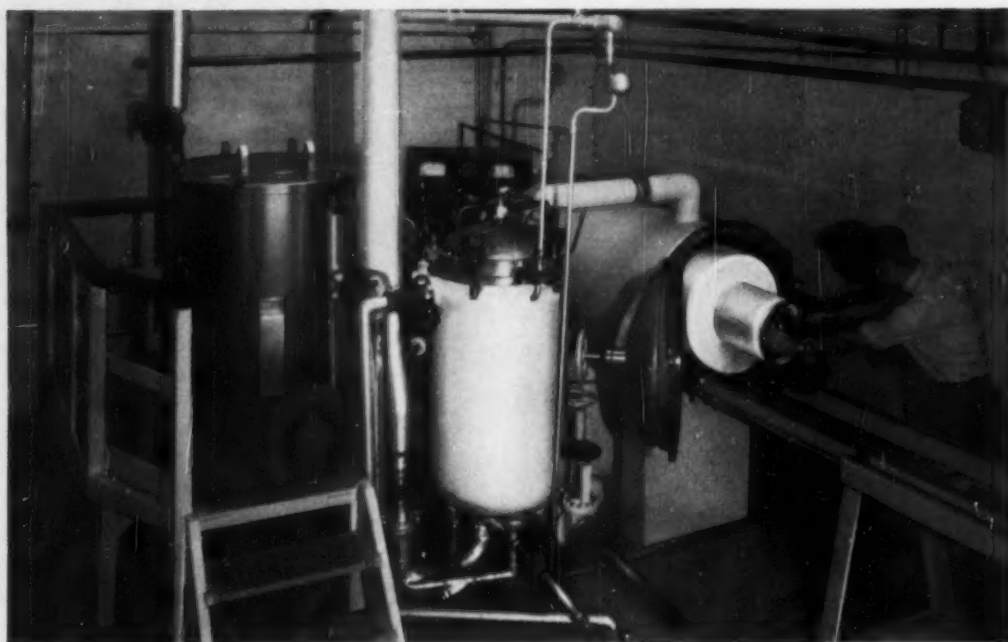
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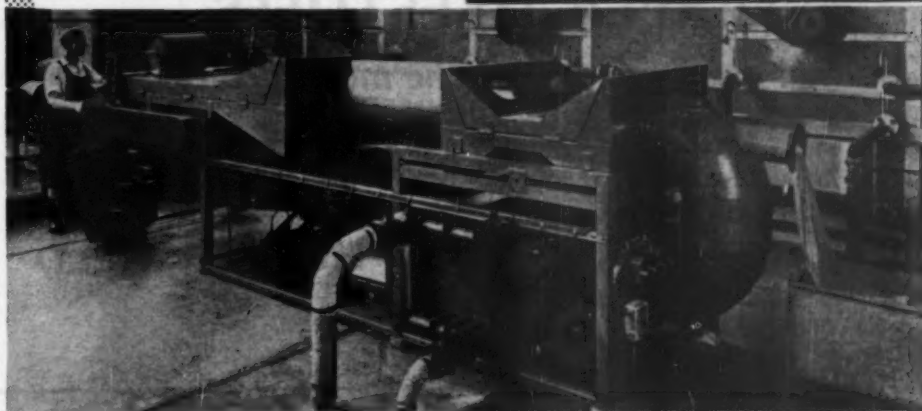
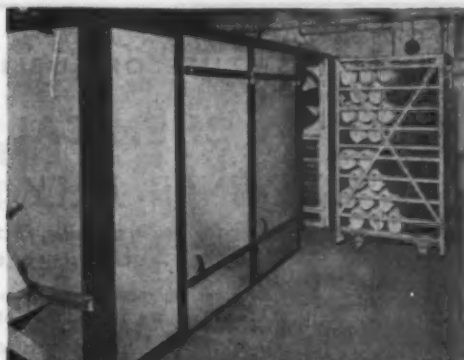
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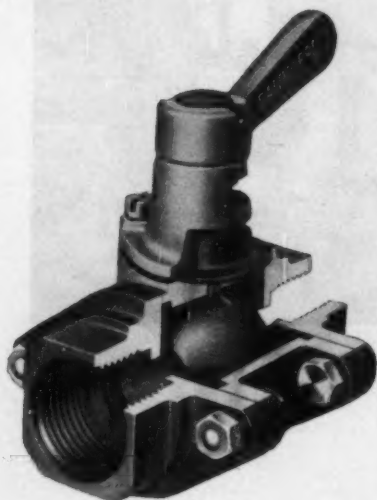
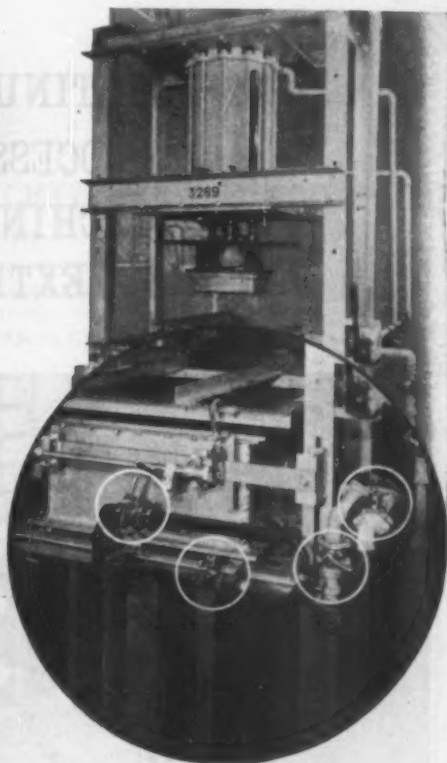
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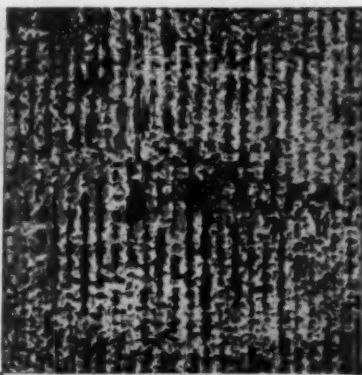
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